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## Sediment Quality Criteria Report for Permethrin

### Phase III: Application of the Pesticide Sediment Quality Criteria Methodology



Prepared for the Central Valley Regional Water Quality Control Board

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## **Disclaimer**

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# Table of Contents

Table of Contents .....	iv
List of Tables .....	vi
List of Figures .....	vi
List of acronyms and abbreviations .....	vii
1 Introduction.....	1
2 Basic information.....	1
3 Physicochemical data.....	2
4 Environmental and metabolic fate .....	3
5 Human and wildlife dietary values .....	3
6 Ecotoxicity data .....	4
7 Data prioritization .....	4
8 Acute criterion calculation.....	4
9 Chronic criterion calculation.....	6
10 Water Quality Effects .....	6
10.1 Bioavailability.....	6
10.2 Mixtures .....	7
10.3 Temperature, pH, and other water quality effects.....	9
11 Comparison of ecotoxicity data to derived criteria.....	9
11.1 Sensitive species .....	9
11.2 Ecosystem and other studies .....	10
11.3 Threatened and endangered species.....	12
12 Harmonization with other environmental media .....	13
12.1 Water.....	13
12.2 Biota.....	13
13 Permethrin Criteria Summary .....	13
13.1 Assumptions, limitations and uncertainties .....	13
13.2 Comparison to existing criteria.....	14
13.3 Permethrin interim criteria statement.....	15

Acknowledgements.....	16
Tables.....	17
Figures.....	27
References.....	28
Appendix – Toxicity Data Summaries for Permethrin .....	A1
Appendix 1.....	A2
Appendix 2.....	A67
Appendix 3.....	A97

## List of Tables

Table 1 Physical-chemical properties of permethrin. ....	17
Table 2 $K_{OC}$ and $K_{DOC}$ geometric mean calculations for permethrin using acceptable values. ....	18
Table 3 Permethrin hydrolysis, photolysis, and biodegradation. ....	19
Table 4 Bioconcentration factors (BCF) for permethrin. ....	20
Table 5 Final acute toxicity data used to calculate permethrin bioavailable sediment quality criteria. ....	22
Table 6 Acceptable acute toxicity data excluded in the data prioritization process for permethrin. ....	23
Table 7 Supplemental acute toxicity studies excluded from permethrin bioavailable sediment quality criteria derivation. ....	24

## List of Figures

Figure 1 Structure of permethrin. ....	27
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## List of acronyms and abbreviations

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ACR	Acute-to-chronic ratio
AF	Assessment factor
AFDM	Ash free dry mass
ASTM	American Society for Testing and Materials
BAF	Bioaccumulation factor
BCF	Bioconcentration factor
BSQC	Bioavailable sediment quality criteria
CAS	Chemical Abstract Service
CDFW	California Department of Fish and Wildlife
CDPR	California Department of Pesticide Regulation
CRWQCB	California Regional Water Quality Control Board, Central Valley Region - CVR
DDT	Dichlorodiphenyltrichloroethane
DOC	Dissolved organic carbon
DOE	United States Department of Energy
DOM	Dissolved organic matter
EC <sub>x</sub>	Concentration that affects x% of exposed organisms
EPA	Environmental Protection Agency
EqP	Equilibrium partitioning
ESG	Equilibrium sediment guideline
HPLC	High performance liquid chromatography
HSDB	Hazardous Substance Data Bank
IUPAC	International Union of Pure and Applied Chemistry
<i>K</i>	Interaction coefficient
<i>K<sub>d</sub></i>	Solid-water partition coefficient
<i>K<sub>DOC</sub></i>	Dissolved organic carbon-water partition coefficient
<i>K<sub>OC</sub></i>	Organic carbon-water partition coefficient (also described as organic carbon-normalized solid-water partition coefficient)
<i>K<sub>OW</sub></i>	Octanol-water partition coefficient
<i>K<sub>x</sub></i>	Interaction coefficient for a synergist/antagonist at concentration x
L	Less relevant/reliable
LC <sub>x</sub>	Concentration lethal to x% of exposed organisms
LL	Less relevant and less reliable
LN	Less relevant and not reliable
LOEC	Lowest observed effect concentration
LR	Less relevant but reliable
MATC	Maximum acceptable toxicant concentration
N	Not relevant/reliable
NOEC	No observed effect concentration
NSTP	National Status and Trends Program

OC	Organic carbon
OECD	Organisation for Economic Co-operation and Development
OM	Organic matter
OPPTS	Office of Prevention, Pesticides and Toxic Substances
PBO	Piperonyl butoxide
R	Relevant/reliable
RL	Relevant but less reliable
RN	Relevant but not reliable
RR	Relevant and reliable
RWQCB	Regional Water Quality Control Board
SMAV	species mean acute value
SMCV	species mean chronic value
SPME	Solid-phase microextraction
SSD	Species sensitivity distribution
SSTT	Spiked-sediment toxicity testing
SQC	Sediment quality criteria
SQG	Sediment quality guideline
SWRCB	State Water Resources Control Board
TES	Threatened and endangered species
UCDM	University of California Davis water quality criteria derivation methodology
UCDSM	University of California Davis sediment quality criteria derivation methodology
US	United States
USEPA	United States Environmental Protection Agency
USFDA	United States Food and Drug Administration
WQC	Water quality criteria

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## **1 Introduction**

Permethrin is a pyrethroid insecticide that has been detected in sediments throughout the Sacramento and San Joaquin Rivers watershed and linked to sediment toxicity in both urban and agricultural drainages (Holmes et al. 2008, Weston et al. 2004). Pyrethroids are widely used in agricultural and urban settings for control of invertebrate pests. The pyrethroid insecticides are hydrophobic compounds that quickly partition to sediments and particulates in the environment and are moderately persistent. These compounds are nerve agents that cause over-excitation of the neurons, leading to paralysis and ultimately death. Aquatic invertebrates are particularly sensitive to pyrethroids because they disrupt osmoregulation (Clark and Matsumura 1982). In addition to lethality, sublethal toxic effects of pyrethroids, such as reduced growth, altered behavior and endocrine disruption effects have also been documented, which may contribute to a decrease in an organism's survival, growth or reproduction (Werner and Moran 2008).

Permethrin sediment criteria are calculated and presented as an illustration of the recently developed University of California, Davis sediment quality criteria derivation methodology (UCDSM) which produces a bioavailable sediment quality criteria (BSQC) (Fojut et al. 2014). Current limitations to the criterion calculation are discussed and rationale is provided as to how to best proceed under such conditions. Acute and chronic water quality criteria (WQC) calculated via the UC Davis water quality criteria derivation methodology (UCDM) are available for permethrin (Fojut et al. 2011, Fojut et al. 2012). The first sections (2 - 5) summarize information that was gathered for the WQC report: basic information about permethrin, physicochemical property data, environmental and metabolic fate, and human and wildlife dietary values. The literature was reviewed for current information not included in these sections and updated where appropriate. Following these introductory sections, sediment exposure data is summarized (sections 6 and 7) and the criteria calculations are described (sections 8 and 9). The remaining sections describe potential water quality effects (section 10) and compare other types of ecotoxicity data to the derived criteria (section 11) and check that the BSQC will not lead to adverse effects in other phases (section 12). Finally, the permethrin BSQC and the major assumptions and limitations inherent in the criteria are summarized (section 13).

## **2 Basic information**

This section summarizes the basic information for permethrin, as identified in the permethrin WQC report (Fojut et al. 2011, Fojut et al. 2012). In the future, if a pesticide has the potential to partition to sediments, it would be most efficient to derive both water and sediment criteria simultaneously to prevent repeated summaries of information that

are relevant to both types of criteria. The chemical structure of permethrin and its stereoisomers is presented in Figure 1.

Permethrin is identified by the following CAS and IUPAC names, and with the following trade names (Fojut et al. 2011):

CAS: (3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate

IUPAC: 3-phenoxybenzyl (RS)-cis-trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate

Chemical Formula:  $C_{21}H_{20}Cl_2O_3$

CAS Number: 52645-53-1

CA DPR Chem Code: 2008

USEPA PC Code: 109701 (formerly 598600)

Trade names: Ambush, Dragnet, Ectiban, Exmin, FMC 33297, FMC 41665, ICI-PP 557, Kafil, Kestrel, NRDC-143, NIA 33297, Niagara 33297, Outflank, Outflank-stockade, Perthrine, Picket, Punce, Pramex, S 3151, SBP-1513, Talcord, WL 43479 (Mackay et al. 2006).

### 3 Physicochemical data

The physicochemical data for permethrin are summarized in Table 1. Calculation of geometric mean values for various physicochemical properties is detailed in the WQC report (Fojut et al. 2011) and not repeated here, with the exception of the organic carbon – water partition coefficient ( $K_{OC}$ ), which has particular relevance to calculation of BSQC. The updated acceptable source data used to calculate the geometric mean of the  $K_{OC}$  and the dissolved organic carbon – interstitial water adsorption coefficient ( $K_{DOC}$ ) are presented in Table 2. The  $K_{OC}$  is used in the UCDSM to estimate interstitial water concentrations from OC-normalized sediment concentrations where necessary. The  $K_{DOC}$  may be used to estimate freely dissolved interstitial water concentrations from total interstitial water concentrations. Studies that determined the permethrin  $K_{OC}$  in marine sediments and marine interstitial waters were excluded in the data sets used to calculate the geometric means, as salt and fresh water data are to be treated separately in the UCDSM.

## 4 Environmental and metabolic fate

Permethrin is a nonpolar compound with low aqueous solubility, high lipid solubility (i.e., octanol-water partition coefficient;  $K_{OW}$ ) and a high  $K_{OC}$  (Table 2). The aqueous insolubility of permethrin predisposes it to partition out of water and sorb with strong affinity to sediment, soil particles, suspended matter and solids in general. Off-site movement of permethrin after application is unlikely unless bound to suspended particles or DOM in runoff water (Gan et al. 2005; Weston et al. 2004). Aquatic toxicity has been shown to decrease as a result of the presence of suspended particles, which have been suggested to limit the bioavailability of pyrethroids (Hill 1989; Muir et al. 1985).

Permethrin is stable to hydrolysis at pH 5 and 7, but very slowly undergoes hydrolysis at pH 9 (242 d; Laskowski 2002) and slowly undergoes photolysis in water (110 d; Laskowski 2002). Permethrin was shown to be more persistent under anaerobic soil conditions (half-life = 197 d, 108 d) compared to aerobic conditions (half-life = 39.5 d, 30 d) (Laskowski 2002; Kegley et al. 2008). Permethrin sediment half-lives ranged from 2 to 13 months at 20°C (Gan et al. 2005). Degradation of permethrin can occur under both biotic (microbe-mediated degradation) and abiotic (i.e., photolysis) conditions (Laskowski 2002; Lee et al. 2004). The degradation half-lives for permethrin are summarized in Table 3.

Permethrin does not strongly bioaccumulate or bioconcentrate in biota, with bioconcentration factors (BCFs) ranging from 8 to 2,800 (Table 4).

## 5 Human and wildlife dietary values

There are no FDA action levels for permethrin (USFDA 2000). There are no food tolerances for human consumption of fish, but there are food tolerances for other meat products; tolerances of 0.05 mg/kg for the meat of poultry and hogs are the lowest recommended tolerances in the permethrin reregistration eligibility decision (USEPA 2006a).

Toxicity data for the mallard duck were used in the permethrin WQC report to assess if the derived criteria would be protective of wildlife (Fojut et al. 2011). The mallard duck toxicity values are also relevant for comparison to the derived BSQC for permethrin and are summarized here. A dietary no-observed effect concentration (NOEC) of 125 mg/kg feed for 23-week old mallard ducks was determined over a 20 week period for the endpoints of hens with regressing ovary, food consumption, and number of eggs laid (Beavers et al. 1992). The lowest-observed effect concentration (LOEC) was determined to be 500 mg/kg in this study.

## 6 Ecotoxicity data

Sixteen original single-species spiked-sediment toxicity tests with permethrin were identified and reviewed. Each study was rated for relevance and reliability. Relevance was rated according to Table 8 of the UCDSM (Fojut et al. 2014). If the study rated relevant (R) or less relevant (L) then it was further evaluated for reliability. The reliability evaluation was based on a combination of documentation and acceptability scores calculated according to Tables 9 and 10 of the UCDSM (Fojut et al. 2014). Studies that were rated relevant or less relevant and reliable or less reliable (RR, RL, LR, or LL) were summarized in the data summary sheets (formatted according to Table 14, Fojut et al. 2014). Copies of completed summaries for all studies are included in the [Appendix](#) of this report. Data rated as acceptable (RR) and used directly in the acute criterion derivation are presented in Table 5. Acute studies that were rated RR but that were excluded in the prioritization process are presented in Table 6, including the reason for data exclusion. There were no chronic toxicity values that rated as RR. Supplemental studies rated as RL, LR or LL are used to evaluate the criteria to check that they are protective of particularly sensitive species and threatened and endangered species (Table 7). Four studies were identified that rated as not relevant (N) and were not used for criteria derivation, but are summarized in Appendix 3.

Mesocosm and field studies evaluated for derivation of the permethrin WQC are also relevant to BSQC derivation for permethrin. Six mesocosm, microcosm and ecosystem (field and laboratory) studies were rated R or L according to Fojut et al. (2011) and are summarized in section 11.2.

## 7 Data prioritization

Multiple toxicity values for permethrin for the same species were reduced to one species mean toxicity value according to procedures described in the UCDSM (section 2.5, Fojut et al. 2014). The final acute data set contains two single species mean acute value (SMAVs) and is shown in Table 5. Acceptable acute data were prioritized and some were excluded for reasons including: more sensitive endpoints were available for the species, and tests conducted at standard conditions are preferred over those conducted at non-standard conditions (Table 6).

## 8 Acute criterion calculation

Two of the five taxa required to construct a species sensitivity distribution were available for permethrin, thus an assessment factor was used to calculate the acute BSQC (section 3.4.1, Fojut et al. 2014). The epibenthic crustacean requirement is represented by

the amphipod *H. azteca*, and the benthic insect category is represented by *C. dilutus*. The three missing taxa are an infaunal invertebrate, a mollusk/amphibian/other unrepresented phylum, and a benthic invertebrate from an unrepresented family.

The acute criterion is calculated by first dividing the lowest SMAV in the acceptable (RR) data set by an assessment factor, which results in an estimate of the 5<sup>th</sup> percentile of the SSD (section 3.5.2, Fojut et al. 2014). The lowest SMAV for permethrin was 2.0 µg/g OC, a 10-d *H. azteca* LC<sub>50</sub> (Table 5). The AF is chosen based on the number of taxa in the data set; the AF for a data set with two taxa requirements is 12 (Table 18, Fojut et al. 2014). Applying the AF provides an estimate of the 5<sup>th</sup> percentile of the species sensitivity distribution. The estimated 5<sup>th</sup> percentile is the recommended acute value, which is divided by two to derive the acute BSQC.

#### Interim Acute BSQC Calculation

Acute value = lowest SMAV ÷ assessment factor

$$= 2.0 \text{ µg/g} \div 12$$

$$= 0.17 \text{ µg/g OC}$$

Interim Acute BSQC = acute value ÷ 2

$$= 0.17 \text{ µg/g OC} \div 2$$

$$= 0.083 \text{ µg/g OC}$$

**Interim Acute BSQC = 0.083 µg/g OC**

$$= 83 \text{ ng/g OC}$$

## 9 Chronic criterion calculation

Chronic toxicity values were not available, thus the acute-to-chronic ratio (ACR) method was used to calculate the chronic criterion (section 3.6.3, Fojut et al. 2014). The lack of chronic sediment toxicity data for permethrin prevents the calculation of an ACR by pairing appropriate acute and chronic spiked sediment toxicity studies. Because an experimental ACR cannot be calculated for permethrin, the chronic criterion is calculated with the default ACR of 11.4 (Table 19, Fojut et al. 2014) and the acute value as follows:

### Interim Chronic BSQC Calculation

$$\text{Chronic BSQC} = \text{acute value} \div \text{ACR}$$

$$= 0.17 \mu\text{g/g OC} \div 11.4$$

$$= 0.015 \mu\text{g/g OC}$$

$$\text{Interim Chronic BSQC} = 0.015 \mu\text{g/g OC}$$

$$= 15 \text{ ng/g OC}$$

## 10 Water Quality Effects

### *10.1 Bioavailability*

Bioavailability is directly incorporated into the UCDSM by using bioavailability-based toxicity values to derive criteria. The rationale for the bioavailability approach to BSQC derivation is discussed in section 1.2.2 (Fojut et al. 2014). The BSQC are expressed OC-normalized sediment concentrations, and may be converted to freely dissolved interstitial water concentrations if desired to compare to interstitial water concentrations. If site-specific partition coefficients are available they can be used to convert between phases (section 1.2.3.1, Fojut et al. 2014). If a site-specific partition coefficient is not available, then the geometric mean of acceptable partition coefficients can be used. To compare the OC-normalized sediment BSQC to relevant aqueous concentrations, the BSQC were converted to interstitial water concentrations using the  $K_{OC}$  of 223,000, which is the geometric mean of 8 values (Table 2). The resulting acute and chronic interstitial concentrations were 0.4 ng/L and 0.07 ng/L, respectively.

## 10.2 Mixtures

In general, additive mixture effects can be incorporated in criteria compliance using the concentration-addition model when it has been established that it is reasonable to assume additivity (section 4.2.1, Fojut et al. 2014). When it is demonstrated or can be assumed that mixture effects will be additive, toxic unit analysis is a simple way to check for compliance as long as there are BSQC available for each compound in the mixture. For non-additive mixture effects, interaction coefficients can be used if ample data are available (section 4.2.2, Fojut et al. 2014). More complex mixtures, involving both synergists and antagonists cannot be incorporated into compliance determination at this time, although some complex models do exist to predict effects in these situations (section 4.2.3, Fojut et al. 2014).

Permethrin often occurs in the environment with other pyrethroid pesticides (Trimble et al. 2009, Werner & Moran 2008), and the presence of chemicals in surface waters is ubiquitous. All pyrethroids have the same toxicological mode of action, and several studies have demonstrated that the toxicity of pyrethroid mixtures is additive and is well-predicted by the concentration addition model (Barata et al. 2006, Brander et al. 2009, Trimble et al. 2009). In a review paper that included derivation of water quality criteria for pyrethroids, including permethrin, Fojut et al. (2012) concluded that additivity of pyrethroid mixture toxicity is well-described in the literature and recommended that the concentration-addition method should be used for compliance determination to account for multiple pyrethroids in a sample. This is also the recommendation to determine BSQC compliance.

Brander et al. (2009) tested mixture toxicity of cyfluthrin and permethrin, and found that the combined toxicity was nearly additive. Although the binary mixture demonstrated slight antagonism, additivity was demonstrated when piperonyl butoxide (PBO) was added. Brander et al. (2009) offered several explanations for the observed antagonism between the two pyrethroids. Permethrin is a type I pyrethroid, and cyfluthrin is a type II pyrethroid, and type II pyrethroids might be able to outcompete type I pyrethroids for binding sites, which is known as competitive agonism; or binding sites may be saturated, so that complete additivity is not observed. They also note that cyfluthrin is metabolized more slowly than permethrin, so cyfluthrin can bind longer, and permethrin may be degraded when binding sites open. PBO may remove this effect because the rate of metabolism of both pyrethroids is reduced in the presence of PBO.

To examine if pyrethroid mixture toxicity is additive with a more comprehensive study design, Trimble et al. (2009) performed sediment toxicity tests with *Hyalella azteca* in three binary combinations: type I-type I (permethrin-bifenthrin), type II-type II (cypermethrin- $\lambda$ -cyhalothrin), and type I-type II (bifenthrin-cypermethrin). The toxicity

of these combinations were predicted with the concentration addition model, with model deviations within a factor of two, indicating that in general, pyrethroid mixture toxicity is additive.

Piperonyl butoxide (PBO) is commonly added to pyrethroid insecticide treatments because it is known to increase the toxic effects of pyrethroids (Weston et al. 2006). Many studies have demonstrated that the addition of PBO at a concentration that would be nonlethal on its own, increases the toxicity of permethrin for fish, insects, crustaceans and mollusks, with interaction coefficients ranging from 1.54-60, as summarized below. Brander et al. (2009) observed *Hyaella azteca* LC<sub>50</sub>s decreased by a factor of 3.5 when a nonlethal concentration of PBO was mixed with permethrin. Paul and Simonin (2006) reported that toxicity to crayfish increased by a factor of 2.1 when testing a formulation that contained 31.28% permethrin and 66% PBO compared to a product that was 92% permethrin (0% PBO) based on the 96-h LC<sub>50</sub>. Paul et al. (2005) reported a significant difference between technical permethrin vs. PBO-synergized permethrin in toxicity to brook trout from 24-96 h and an interaction coefficient (K) of 2.9. The addition of a nonlethal concentration of PBO reduced the LC<sub>50</sub> of permethrin to snails with a K of 60 at 96 h (Singh & Agarwal 1986).

Permethrin toxicity with and without PBO was tested with mosquitoes by Hardstone et al. (2007, 2008) with a permethrin-susceptible strain, resulting in an K of 1.54. Kasai et al. (1998) also did experiments with *Culex quinquefasciatus* mosquitoes and demonstrated that a nonlethal concentration of 0.5 mg/L PBO decreased the LC<sub>50</sub> of permethrin from 4 ug/L to 0.44 ug/L in a permethrin-susceptible strain. Xu et al. (2005) tested permethrin toxicity to *C. quinquefasciatus* with and without PBO and reported a K of 4.5 for a permethrin-susceptible strain. Paul et al. (2006) tested *Aedes aegypti* mosquitoes and reported a K for permethrin and PBO of 11. While many studies report interaction coefficients for synergism of PBO, none of them reported Ks for multiple PBO concentrations, so a relationship between PBO concentration and K cannot be determined for any given species. Consequently, it is not possible to quantify this non-additive toxicity and there is no accurate way to account for this interaction in compliance determination.

Corbel et al. (2003) tested the toxicity of permethrin in combination with propoxur, a carbamate, with mosquito larvae and found that equitoxic mixtures of the two chemicals demonstrated synergism, which the authors propose is due to the complementary modes of action acting on different parts of the nervous system. Zhang et al. (2010) tested mixtures of permethrin with the organophosphates dichlorvos or phoxim with zebrafish and reported that the toxicity of binary combinations was additive.

No studies on aquatic organisms were found in the literature that could provide a quantitative means to consider mixtures of permethrin with other classes of pesticides.



Although there are examples of non-additive toxicity for permethrin and other chemicals, a multispecies interaction coefficient is not available for any chemical with permethrin, and therefore the concentrations of non-additive chemicals cannot be used for criteria compliance (section 3-5.2.2, TenBrook et al. 2009).

### ***10.3 Temperature, pH, and other water quality effects***

The effects of temperature, pH, and other water quality parameters on the toxicity of permethrin were examined to determine if these are described well enough in the literature to incorporate into BSQC compliance (section 4.3, Fojut et al. 2014). The effects of temperature and pH on pyrethroid toxicity were discussed previously in the permethrin WQC report (Fojut et al. 2011) and this discussion is also applicable to sediment toxicity. To summarize, there is an inverse relationship between temperature and the toxicity of pyrethroids (Miller and Salgado 1985; Werner and Moran 2008), and this relationship is likely the result of an increased sensitivity of the organism's sodium channel at lower temperatures (Narahashi et al. 1998). Pyrethroid contaminated sediments were more than twice as toxic to *H. azteca* when tested at 18°C compared to 23°C in the laboratory (Weston et al. 2008). Weston et al. (2008) found that temperatures required in standard methods are likely higher than environmental temperatures and toxicity may be underestimated as a result of colder habitats. These results are not directly applicable for use in BSQC compliance because environmental samples were used, instead spiked sediment toxicity tests.

Despite the known effect of temperature on pyrethroid toxicity, there is not enough information to incorporate temperature effects in BSQC or compliance at this time. Also, no studies could be found that addressed pH or other water quality effects on permethrin toxicity in sediment or interstitial water. As a result, information is insufficient at this time to be able to incorporate the effects of water quality parameters into BSQC compliance.

## **11 Comparison of ecotoxicity data to derived criteria**

### ***11.1 Sensitive species***

A data comparison was conducted to assess if the derived criteria for permethrin are protective of the most sensitive species. In the following, the derived BSQC are compared to toxicity values for the most sensitive species in both the acceptable (RR) and supplemental (RL, LR, LL) data sets (section 5.1, Fojut et al. 2014).

The lowest reported acute sediment toxicity value in the RR data set is a 10-d LC<sub>50</sub> of 2.0 (1.5-2.6) µg/g OC for *H. azteca* (Picard 2010a; Table 5) and was used directly in the criteria derivation. The lowest acute toxicity value in the supplemental data set is a 10-d LC<sub>50</sub> of 0.829 µg/g OC *Ampelisca abdita*, which is an estuarine species (Anderson et al. 2008; Table 7). The interim acute BSQC of 0.083 µg/g OC is a factor of 10 below this toxicity value and the BSQC very protective based on this toxicity value.

Some of the SSTT studies used to calculate the acute BSQC also reported NOEC or LOEC values for 10-day exposures. Since 10-day NOEC/LOECs do not meet the requirements for inclusion in the acute data set (which requires LC/EC<sub>50</sub>s) or the chronic data set (which requires ≥ 28-d full or partial life cycle tests), these values were not used for derivation of BSQC, but are compared to the derived BSQC. The lowest MATC reported for *H. azteca* is 0.42 µg/g OC based on a 10-d growth endpoint (Picard et al. 2010a). This value is approximately 5-fold higher than the interim acute BSQC, which indicates that the interim acute BSQC very protective.

There are SSTT data for chronic exposure durations (20-d and 58-d) for the midge *Chironomus dilutus* (Table 7). The study did not report NOEC and LOEC values, instead LC<sub>5</sub>, EC<sub>5</sub>, LC<sub>50</sub>, and EC<sub>50</sub> values were reported. The lowest toxicity value for these chronic exposure durations is a 58-day EC<sub>5</sub> for reproduction of 0.009 ± 0.008 µg/g OC (Du et al. 2013). This value is below the interim chronic BSQC of 0.015 µg/g OC by a factor of 1.7. EC<sub>5</sub> values are not used as chronic toxicity values in the UCDSM because these values may be more influenced by regression fitting parameters than by the exposure data. In this particular study the 5% effect concentrations are extrapolated rather than interpolated, meaning these effects were not observed in the treatments at these levels. The interim BSQC will not be adjusted downward based on interpolated toxicity values because these values are highly uncertain, which is indicated by the high relative standard deviation of this toxicity value.

## ***11.2 Ecosystem and other studies***

In this section, the derived permethrin criteria are compared to acceptable laboratory, field, or semi-field multispecies studies (rated R or L), to determine if the criteria will be protective of ecosystems. Thirteen studies describing effects of permethrin on mesocosm, microcosm and model ecosystems were identified and rated for reliability according to the methodology (Table 11, Fojut et al. 2014). Six of the studies were rated as less reliable (L; Conrad et al. 1999, Coulon 1982, Lutnicka et al. 1999, Poirier & Surgeoner 1988, Werner & Hilgert 1992, Yasuno et al. 1988) and are used as supporting data. Seven studies rated as not reliable (N) and are not discussed in this report (Feng et al. 2009, Helson et al. 1986, 1993, Jensen et al. 1999, Milam et al. 2000, Mulla et al. 1978, Soltani et al. 2012). None of the studies report a community NOEC to which the

calculated chronic criterion may be compared. All of the reported aqueous test concentrations were significantly higher than the UCDM chronic WQC of 0.002 µg/L, with concentrations ranging from 0.02-100 µg/L, and all studies were conducted with formulations of permethrin. Only one study reported sediment concentrations; Conrad et al. (1999) reported effects at concentrations ranging from 0.6-9.4 µg/g OC, and no significant effects at 0.17 µg/g OC. All of these concentrations are above the interim chronic BSQC of 0.015 µg/g OC.

Two studies reported increased invertebrate drifting after exposure to permethrin. Werner & Hilgert (1992) reported residues of 0.02-0.14 µg/L permethrin had drifted into an Alaskan stream after spruce trees were sprayed, and drifting of aquatic invertebrates (Chironomidae, ephemeropteran and, trichopteran larvae) significantly increased after the treatment, but trout fry, periphyton, and benthic invertebrates were not affected. Poirier & Surgeoner (1988) exposed various aquatic invertebrates to flowing stream water in constructed troughs with 1-h application of a permethrin formulation (Ambush® EC) at 7-10 concentrations. LC<sub>50</sub>s were reported for six invertebrates ranging from 2.0-7.1 µg/L, although invertebrate drift occurred at all concentrations greater than 0.5 µg/L permethrin. Lutnicka *et al.* (1999) also set up model riverine systems containing sediment and moderately contaminated river water and stocked them with lab cultures of water-thyme (*Elodea*), snails and carp (*Cyprinus carpio*). Permethrin was added at two concentrations (4 and 20 µg/L) and snails and water-thyme were both adversely affected at both concentrations.

Pond exposures also demonstrated adverse effects on various aquatic invertebrates, while fish were unaffected. Yasuno et al. (1988) tested permethrin in enclosures set in a pond and studied the effects on the naturally occurring species of the pond, including phytoplankton and various types of zooplankton. Daphnids and their main predator, *Chaoborus*, were both seriously affected by permethrin, and both populations disappeared and did not seem to recover after two treatments of permethrin spaced 18 d apart at a nominal treatment level of 1.5 µg/L. Coulon (1982) tested the Ambush® formulation and reported no mortality of catfish reared in ponds at any of the exposures (0.53-11.09 µg/L measured at 24 h), but aquatic insects were temporarily eliminated. The insects re-inhabited the ponds 10-d post-application. Conrad et al. (1999) dosed small artificial ponds with permethrin and conducted bioassays with chironomids and also observed aquatic invertebrate abundances. The nominal aqueous concentrations of permethrin dosing were 1, 10, 50, and 100 µg/L and the corresponding sediment concentrations at these doses were 4, 14, 180, and 217 ng/g, respectively, and the corresponding pore water concentrations were 1.7, 5.9, 76, and 91.7 ng/L, respectively. The ponds were dosed with the formulation called Picket®. The field exposure data were compared to laboratory sediment toxicity tests with *Chironomus riparius*. The

chironomid response in the ponds of reduced larval density and adult emergence was not predicted by bulk sediment chemistry, sediment toxicity tests or laboratory bioassay results – all three measurements underestimated the acute effects. Acute effects were recorded in ponds dosed at 10, 50, and 100 µg/L. The organic carbon-normalized sediment concentrations for these doses are 0.6, 7.8, and 9.4 µg/g OC based on 9.64% OC in the sediment. Toxicity to *C. riparius* in the field was best predicted by acute water-only toxicity test data, indicating that the primary exposure route is via the water column. This study supports the use of the freely dissolved fraction for water quality criteria compliance and affirms the relevance of water quality criteria for highly sorptive pesticides like pyrethroids.

### ***11.3 Threatened and endangered species***

In this section, the derived criteria for permethrin are compared to toxicity values for threatened and endangered species to ensure that the criteria will be protective of these species (section 5.2, TenBrook et al. 2009). Current records of state and federally listed threatened and endangered animal species in California were obtained from the CDFW web site (<http://www.dfg.ca.gov/biogeodata/cnddb/pdfs/TEAnimals.pdf>; CDFW 2013).

No listed threatened or endangered species are included in the acceptable and supplemental data sets used for permethrin BSQC derivation (Table 5 through Table 7). Similar to the WQC report (Fojut et al. 2011), no data were found for effects of permethrin on federally endangered crustaceans and insects, or acceptable surrogates (i.e., in the same family). In the WQC report, the lowest toxicity value for a threatened or endangered species was an LC<sub>50</sub> of 1.58 (1.1-2.2) µg/L for *Oncorhynchus clarki henshawi* (Fojut et al. 2011). The acute and chronic BSQC were converted to interstitial concentrations of 0.4 ng/L and 0.07 ng/L, respectively, to compare to this aqueous value. The acute and chronic BSQC are far below this toxicity value. Based on the little available data, there is no evidence that the interim acute and chronic permethrin BSQC will be under-protective of threatened or endangered species but this assessment lacks chronic data and data for crustaceans and insects, which are considered the most sensitive species.

No single-species plant studies were found in the literature for use in criteria derivation, so no estimation could be made for plants on the state or federal endangered, threatened or rare species lists. There are also no aquatic plants listed as state or federal endangered, threatened or rare species so they are not considered in this section. Based on the available data and estimated values for animals, there is no evidence that the calculated acute and chronic criteria will be underprotective of threatened and endangered species.

## **12 Harmonization with other environmental media**

### ***12.1 Water***

The BSQC were converted from OC-normalized sediment concentrations to interstitial water concentrations to compare them to existing water quality criteria. The  $K_{OC}$  of 223,000, which is the geometric mean of 8 values (Table 2), was used as the partition coefficient. The resulting acute and chronic BSQC interstitial concentrations were 0.4 ng/L and 0.07 ng/L, respectively. The permethrin acute and chronic WQC are 10 ng/L and 2 ng/L, respectively, which are above the BSQC concentrations. Therefore, if the BSQC were attained it would be unlikely that the WQC would be exceeded due to desorption from sediment, if equilibrium conditions are assumed.

### ***12.2 Biota***

Based on the mean log  $K_{OW}$  of permethrin of 6.3 and its molecular weight of 391.29 g/mol, permethrin has the potential to bioaccumulate (section 6.2, Fojut et al. 2014). In the UCDM WQC report, the accumulation of permethrin in food items to levels that are known to cause harm to their predators was examined to ensure WQC were protective (Fojut et al. 2011). To assess the risk of secondary poisoning, a calculated BAF (28,000 L/kg, Fojut et al. 2011) and the NOEC values for mallard (125 mg/kg feed; Beavers et al. 1992) were used to roughly estimate water concentrations that would equate to no-effect levels for consumption of fish by terrestrial wildlife (Fojut et al. 2011). The estimated NOEC was 4.46 µg/L for mallard duck. The chronic permethrin WQC and interstitial water BSQC (2 ng/L and 0.07 ng/L, respectively) are below this value, indicating that compliance with the BSQC should not conflict with other efforts to protect wildlife from permethrin exposure.

## **13 Permethrin Criteria Summary**

### ***13.1 Assumptions, limitations and uncertainties***

The assumptions, limitations and uncertainties involved in criteria derivation should be available to inform environmental managers of the accuracy and confidence in the derived criteria. This section summarizes any data limitations that affected the procedure used to determine the final permethrin criteria.

For the permethrin interim acute BSQC, a major limitation was the lack of acute SSTT data for freshwater species other than *H. azteca* and *C. dilutus*. Three of the five

taxa requirements of the UCDSM were not met, and as such, an assessment factor approach was used to calculate the acute BSQC. Similarly, the major limitation for the permethrin interim chronic BSQC derivation was the lack of freshwater species in the chronic toxicity data set. None of the five taxa requirements were met, which precluded the use of a SSD; therefore, an ACR was used to derive the chronic criterion. Since no acceptable experimental ACRs were available for permethrin in the literature, the default ACR of 11.4 was used. Particularly of concern was the lack of chronic data for *H. azteca*, which was the most sensitive species in the acute toxicity data set. Uncertainty cannot be quantified for either the acute or chronic criteria because they were not derived with a SSD.

To compare the OC-normalized sediment BSQC to relevant aqueous concentrations, the BSQC were converted to interstitial water concentrations using the  $K_{OC}$  of 223,000, which is the geometric mean of 8 values (Table 2). The resulting acute and chronic interstitial concentrations were 0.4 ng/L and 0.07 ng/L, respectively.

As concluded in the permethrin WQC report, increased permethrin toxicity as a result of lower temperatures still cannot be accounted for quantitatively (Fojut et al. 2011). An additional safety factor is not recommended to adjust criteria at this time but environmental managers should keep this factor in mind if derived criteria are not protective in colder water bodies.

Although greater than additive effects have been observed for mixtures of pyrethroids and PBO, there is insufficient data to account for this interaction in compliance determination. This is a significant limitation because formulations that contain both pyrethroids and PBO are available on the market. When additional highly rated data are available, the criteria should be recalculated to incorporate new research.

### ***13.2 Comparison to existing criteria***

To date, no USEPA sediment criteria or benchmarks are available for permethrin. The USEPA proposes an EqP-based approach, through which, the chronic WQC is used to predict the corresponding sediment concentration using the  $K_{OC}$  (Di Toro et al. 2002). The lowest SMAV in the acceptable sediment data set was converted to an interstitial water concentration to compare it to existing WQC. The lowest SMAV in the RR data set of 2.0 µg/g OC, a 10-d *H. azteca* LC<sub>50</sub> (Table 5), was converted to an interstitial concentration of 9 ng/L using the geometric mean of  $K_{OC}$  of 223,000. This value is compared to the chronic WQC for permethrin of 2 ng/L, which is a factor of 4.5 lower than the lowest SMAV. Thus, the chronic WQC would be very protective of short-term effects from sediment-associated permethrin. The lowest EC<sub>50</sub> for a chronic exposure duration is the 58-d *C. dilutus* reproduction EC<sub>50</sub> of 0.039 µg/g OC (Table 7), which was converted to an interstitial concentration of 0.17 ng/L. The chronic WQC of 2 ng/L is

approximately a factor of 12 higher than the lowest SMCV. Thus, the chronic WQC may not be protective of long-term effects from sediment-associated permethrin.

### ***13.3 Permethrin interim criteria statement***

The interim criteria statement is:

Aquatic life should not be affected unacceptably if the 28-day average concentration of permethrin does not exceed 0.083 µg/g OC (83 ng/g OC) in sediment more than once every three years on average and if the 10-day average concentration does not exceed 0.015 µg/g OC (15 ng/g OC) in sediment more than once every three years on average.

Although the criteria were derived to be protective of aquatic life in the Sacramento and San Joaquin Rivers, these criteria would be appropriate for any freshwater ecosystem in North America, unless species more sensitive than are represented by the species examined in the development of the present criteria are likely to occur in the ecosystems of interest.

The final acute criterion was derived using the AF procedure and the acute data used in criteria calculation are shown in Table 5. The chronic criterion was derived by use of a default ACR.

## **Acknowledgements**

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# Tables

Table 1 Physical-chemical properties of permethrin.

Property	Permethrin
Chemical formula	$C_{21}H_{20}Cl_2O_3$
Chemical Abstract Service (CAS) number	52645-53-1
California Department of Pesticide Regulation chemical code	2008
Classification	EPA toxicity class II or III <sup>a</sup>
Molecular weight	391.29
Density (g/mL)	1.19-1.27
Water solubility (mg/L)	0.0057 (geomean, n=2)
Melting point (°C)	36.4 (geomean, n=2)
Vapor pressure (Pa)	$3.74 \times 10^{-6}$ (geomean, n=4)
Henry's law constant ( $K_H$ ) ( $Pa\ m^3\ mol^{-1}$ )	0.12 (geomean, n=2)
Log-normalized organic carbon-water partition coefficient (log $K_{OC}$ )	5.35 (geomean, n=8)
Log-normalized octanol-water partition coefficient (log $K_{OW}$ )	6.3 (geomean, n=2)

<sup>a</sup>EXTOXNET 1995

Table 2  $K_{OC}$  and  $K_{DOC}$  geometric mean calculations for permethrin using acceptable values.

$K_{OC}$	$K_{DOC}$	Reference
86,500 (mean, n=76)	-	Laskowski 2002
63,100	-	Meylan et al. 1992
100,000	-	Wauchope et al. 1992
127,000 85,000 91,000	-	Zhou & Rowland 1997
2,350,000 (cis) 4,870,000 (trans)	21,350,000 (cis) 44,780,000 (trans)	Cui & Gan 2013
-	69,000 $\pm$ 1,000 (cis) 68,000 $\pm$ 4,000 (trans)	Delgado-Moreno et al. 2010
-	240,000 (cis) 140,000 (trans) 80,000 (cis) 40,000 (trans) 410,000 (cis) 250,000 (trans) 1,150,000 (cis) 720,000 (trans)	Bondarenko & Gan 2009
-	160,000 (cis) 160,000 (trans) 160,000 (cis) 180,000 (trans)	Liu et al. 2004
-	69,000 $\pm$ 18,000 126,000 $\pm$ 30,000 54,000 $\pm$ 120,000 83,000 $\pm$ 42,000	Yang et al. 2006
<b>223,000</b>	<b>253,000</b>	<b>Geometric mean</b>

Table 3 Permethrin hydrolysis, photolysis, and biodegradation.

	<b>Half- life (d)</b>	<b>Water</b>	<b>Temp (°C)</b>	<b>pH</b>	<b>Reference</b>
Hydrolysis	Stable	Sterile, buffered	25	5	Laskowski 2002
	Stable	Sterile, buffered	25	7	Laskowski 2002
	242	Sterile, buffered	25	9	Laskowski 2002
Aqueous Photolysis	110	NR	25	5	Amos & Donelan 1987, Laskowski 2002
Soil Biodegradation (aerobic)	39.5	3 soil types (n=8)	16-25	n/a	Laskowski 2002

Table 4 Bioconcentration factors (BCF) for permethrin.  
FT: flow-through, S: static.

Species	BCF (L/kg)	Exposure	Reference
<i>Anabaena</i> (cyanobacteria)	57-813	S, 5 d	Kumar <i>et al.</i> 1988
<i>Aulosira fertilissima</i> (cyanobacteria)	46-2373	S, 5 d	Kumar <i>et al.</i> 1988
<i>Chironomus dilutus</i>	87.2	S, 96 h, 23°C	Harwood <i>et al.</i> 2009
<i>Chironomus tentans</i>	8-166	S, 24 h, water-sediment system	Muir <i>et al.</i> 1985
<i>Crassostrea virginica</i>	1900	FT	Schimmel <i>et al.</i> 1983
<i>Cyprinodon variegatus</i>	290-620	FT, 28 d	Hansen <i>et al.</i> 1983
<i>Daphnia magna</i>	<b>BAF</b> sed 1: 951 sed 2: 808 sed 3: 1,071 sed 4: 1,045	S, 24 h, water-sediment system	Yang <i>et al.</i> 2006
<i>Helisoma trivolvis</i> (snail)	800	FT, 30 d	Spehar <i>et al.</i> 1983
<i>Hydrophilus</i> spp. (water scavenger beetle)	4.10 L/g	S, 6 h	Tang & Siegfried 1996
<i>Hydropsyche</i> & <i>Chematapsyche</i> spp. (caddisfly)	30.4 L/g	S, 6 h	Tang & Siegfried 1996
<i>Ishnura</i> & <i>Enallagma</i> spp. (damselfly)	6.87 L/g	S, 6 h	Tang & Siegfried 1996
<i>Lepomis macrochirus</i>	558	FT, 28 d	Burgess 1989
<i>Lepomis macrochirus</i>	681	FT, 28 d	Tullman 1989
<i>Lumbriculus variegatus</i>	1466	SR, 14 d	You <i>et al.</i> 2009
<i>Oncorhynchus mykiss</i>	328-631	FT, 4 d	Muir <i>et al.</i> 1994
<i>Pimephales promelas</i>	2800	FT, 30 d	Spehar <i>et al.</i> 1983
<i>Salmo salar</i>	14-73 L/g	S, 96 h	McLeese <i>et al.</i> 1980
<i>Salmo salar</i>	55	S, 96 h	Zitko <i>et al.</i> 1977

<i>Simulium vittatum</i> (black fly)	17.9 L/g	S, 6 h	Tang & Siegfried 1996
<i>Stenacron</i> spp. (mayfly)	23.6 L/g	S, 6 h	Tang & Siegfried 1996
<i>Tetrahymena</i> <i>pyriformis</i> (protozoa)	70-1110	2-12 h	Bhatnagar <i>et al.</i> 1988

Table 5 Final acute toxicity data used to calculate permethrin bioavailable sediment quality criteria.  
All studies were rated relevant and reliable (RR).

Species	Common name	Family	Duration (d)	Temp (°C)	Endpoint	Age/ size	Sediment LC/EC <sub>50</sub> (95% CI) (µg/g OC)	% OC	Ref
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Growth (AFDM)	3 <sup>rd</sup> instar	5.2 (4.3-6.2)	2.1	a
“	“	“	10	23	Growth (AFDM)	3rd instar	27.4 (14.4-60.9)	0.69	b
<i>Chironomus dilutus</i>					Geometric mean		<b>11.9</b>		
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23±1	Growth	8 d	<b>2.0</b> (1.5-2.6)	2.3	c

LC<sub>50</sub> = exposure concentration lethal to 50% of a test population, EC<sub>50</sub> = exposure concentration that causes effect in 50% of a test population, CI: confidence interval, OC = organic carbon, Chir. = Chironomidae, AFDM = ash free dry mass, Hyal. = Hyalellidae.

<sup>a</sup>Picard et al. 2010b, <sup>b</sup>Maul et al. 2008, <sup>c</sup>Picard et al. 2010a.

Table 6 Acceptable acute toxicity data excluded in the data prioritization process for permethrin.  
All studies were rated relevant and reliable (RR).

Species	Common name	Family	Duration (d)	Temp (°C)	Endpoint	Age/ size	Sediment LC/EC <sub>50</sub> (95% CI) (µg/g OC)	% OC	Ref	Excl
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	13	Survival	3 <sup>rd</sup> instar	9.16 (3.48-18.5)	0.69	a	1
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Survival	3 <sup>rd</sup> instar	20.5 (17.6-23.8)	2.1	b	
“	“	“	10	23	Survival	3 <sup>rd</sup> instar	27.4 (19.0-37.8)	0.69	a	
“	“	“	10	23	Survival	3 <sup>rd</sup> instar	24.5 (5.7-58.9)	0.69	c	
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Geometric mean		24.0			2
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Growth (IGR)	3 <sup>rd</sup> instar	27.2 (15.4-58.3)	0.69	c	2
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23±1	Survival	8 d	2.6 (2.3-2.9)	2.3	d	
“	“	“	10	23±0.1	Survival	7-10 d	23.9 (23.4-24.4)	2.0	e	
<i>Hyaella azteca</i>	Amphipod	Hyal.			Geometric mean		7.9			2

LC<sub>50</sub> = exposure concentration lethal to 50% of a test population, EC<sub>50</sub> = exposure concentration that causes effect in 50% of a test population, CI: confidence interval, OC = organic carbon, Ref = reference, Excl. = reason for exclusion, Chir. = Chironomidae, IGR = instantaneous growth rate, AFDM = ash free dry mass, Hyal. = Hyalellidae.

<sup>a</sup>Harwood et al. 2009, <sup>b</sup>Picard et al. 2010b, <sup>c</sup>Maul et al. 2008, <sup>d</sup>Picard et al. 2010a, <sup>e</sup>Weston & Jackson 2009

<sup>1</sup>Data at standard temperature available.

<sup>2</sup>Data with more sensitive (standard) endpoint available

Table 7 Supplemental acute toxicity studies excluded from permethrin bioavailable sediment quality criteria derivation. Studies rated less relevant and/or less reliable: RL, LR, or LL.

Species	Common name	Family	Duration (d)	Temp (°C)	Endpoint	Age/size	LC/EC <sub>50</sub> (µg/g OC)	% OC	MATC (ng/L)	Ref	Rating, Excl.
<i>Ampelisca abdita</i>	Amphipod	Ampeliscidae	10	20	Survival	NR	0.829	0.78	-	a	LL, 1,3
<i>Ampelisca abdita</i>	Amphipod	Ampeliscidae	10	20	Survival	NR	1.33	0.78	-	a	LL, 1,3
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Immobility	3 <sup>rd</sup> instar	11.5 (7.8-15.4)	0.69	-	b	LR, 2
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Survival	3 <sup>rd</sup> instar	17.6	0.98	-	c	LR, 2
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Survival	3 <sup>rd</sup> instar	5.2	0.98	-	c	LR, 2
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Survival	3 <sup>rd</sup> instar	12	0.98	-	c	LR, 2
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	20	Survival	3 <sup>rd</sup> instar	21.9	9.64	-	d	LL, 4
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Survival	3 <sup>rd</sup> instar	-	2.1	7.7	g	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	20	23	Survival	<24h	LC <sub>5</sub> : 0.143±0.02 2	1.6	-	i	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	58	23	Survival	<24h	LC <sub>5</sub> : 0.075	1.6	-	i	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	20	23±1	Survival	<24h	1.83±1.13	1.60±0.14	-	i	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	58	23±0	Survival	<24h	1.20±0.55	1.60±0.14	-	i	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Growth (AFDM)	3 <sup>rd</sup> instar	EC <sub>20</sub> : 12.6	0.69	-	b	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Growth (AFDM)	3 <sup>rd</sup> instar	-	0.69	53.2	b	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Growth (IGR)	3 <sup>rd</sup> instar	EC <sub>20</sub> : 13.7	0.69	-	b	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	10	23	Growth (IGR)	3 <sup>rd</sup> instar	-	0.69	53.2	b	6



Species	Common name	Family	Duration (d)	Temp (°C)	Endpoint	Age/size	LC/EC <sub>50</sub> (µg/g OC)	% OC	MATC (ng/L)	Ref	Rating, Excl.
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	20	23±1	Growth	<24h	1.09±0.53	1.60±0.14	-	i	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	20	23	Growth	<24h	EC <sub>5</sub> : 0.034±0.006	1.6	-	i	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	58	23±1	Emergence	<24h	0.838±0.77	1.60±0.14	-	i	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	58	23	Emergence	<24h	EC <sub>5</sub> : 0.016±0.008	1.6	-	i	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	58	23 ± 1	Reproduction	<24h	0.039 ± 0.105	1.60 ± 0.14	-	i	6
<i>Chironomus dilutus</i>	Midge (Insect)	Chir.	58	23	Reproduction	<24h	EC <sub>5</sub> : 0.009±0.008	1.6	-	i	6
<i>Eohaustorius estuarius</i>	Amphipod	Haustoridae	10	15	Survival	NR	18.3	0.78	-	a	LL, 1,3
<i>Eohaustorius estuarius</i>	Amphipod	Haustoridae	10	15	Survival	NR	18.8	0.78	-	a	LL, 1,3
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Growth	6-10 d	-	1.4	9.07	e	LL, 4
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Growth	6-10 d	-	1.1	9.07	e	LL, 4
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Growth	6-10 d	-	2.3	0.42	h	6
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Survival	6-10 d	17.87	1.4	-	e	LL, 4
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Survival	6-10 d	11.1	1.1	-	e	LL, 4
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Survival	6-10 d	3.51	6.5	-	e	LL, 4
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Survival	7-10 d	14.2	1.87	-	f	RL, 5
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Survival	7-10 d	21.3	1.87	-	f	RL, 5
<i>Hyaella</i>	Amphipod	Hyal.	10	23	Survival	1-2 weeks	7.59	0.98	-	c	LR, 2

Species	Common name	Family	Duration (d)	Temp (°C)	Endpoint	Age/size	LC/EC <sub>50</sub> (µg/g OC)	% OC	MATC (ng/L)	Ref	Rating, Excl.
<i>azteca</i>											
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Survival	1-2 weeks	6.39	0.98	-	c	LR, 2
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Survival	1-2 weeks	6.39	0.98	-	c	LR, 2
<i>Hyaella azteca</i>	Amphipod	Hyal.	10	23	Survival	6-10 d	-	2.3	1.6	h	6

LC<sub>50</sub> = exposure concentration lethal to 50% of a test population, EC<sub>50</sub> = exposure concentration that causes effect in 50% of a test population, CI: confidence interval, OC = organic carbon, Ref = reference, Excl. = reason for exclusion, Chir. = Chironomidae, Hyal. = Hyalellidae.

<sup>a</sup>Anderson et al. 2008, <sup>b</sup>Maul et al. 2008, <sup>c</sup>Ding et al. 2013, <sup>d</sup>Conrad et al. 1999, <sup>e</sup>Amweg et al. 2005, <sup>f</sup>Amweg et al. 2006, <sup>g</sup>Picard 2010b, <sup>h</sup>Picard 2010a, <sup>i</sup>Du et al. 2013.

<sup>1</sup>Saltwater

<sup>2</sup>Control response not reported or not acceptable

<sup>3</sup>Effects reported as > value

<sup>4</sup>Toxicity value not based on measured bioavailable concentration

<sup>5</sup>Low reliability score

<sup>6</sup>Toxicity value does not fit definition (section 2.1.3.1.2, Fojut et al. 2014)

## Figures

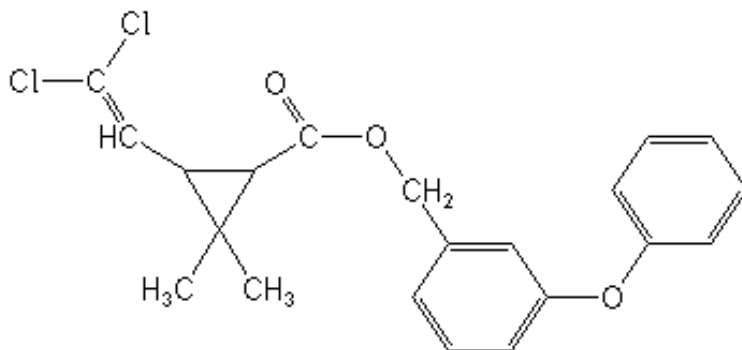


Figure 1 Structure of permethrin.  
(<http://www.alanwood.net/pesticides/permethrin.html>)

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# **Appendix – Toxicity Data Summaries for Permethrin**

Interim Bioavailable Sediment Quality Criteria Report for Permethrin

Appendix

Toxicity data summary sheets



# *Appendix 1*

Studies rated RR

## Toxicity Data Summary

### *Chironomus dilutus*

Du J, Pang J, and You J (2013) Bioavailability-based chronic toxicity measurements of permethrin to *Chironomus dilutus*. Environ Toxicol Chem 32(6):1403–1411

#### Relevance

Score: 100

Rating: R

#### Reliability

Score: 78.5

Rating: R

<i>C. dilutus</i>	<b>Du et al. 2013</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test method cited	EPA 2000	
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	Newly hatched midge larvae, <24h old	
Source of organisms	Lab culture	Guangzhou Institute of Geochemistry, Chinese Academy of Sciences
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	

<i>C. dilutus</i>	<b>Du et al. 2013</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Animals randomized?	Not reported	<b>Accept. points</b>
Test vessels randomized?	Not reported	<b>Accept. points</b>
Test duration	20d and 58d	
Effect 1	20d & 58d - Survival	
Control response 1	20d: 78±5.8% 58d: 68±5.8%	
Effect 2	20d - Growth (total ash free dry wt. per midge)	
Control response 2	20d: 0.75±0.19 mg/midge	
Effect 3	Cumulative Emergence	
Control response 3	70%	Estimated from figure 2.
Effect 4	Rate of Emergence	
Control response 4	Not reported	
Effect 5	Reproduction: Sex ratio	
Control response 5	Not reported	
Effect 6	Reproduction: Eggs per female	
Control response 6	850	Estimated from Figure 3A
Effect 7	Reproduction: Eggs per replicate	
Control response 7	5363±3245	
Effect 8	Reproduction: Egg hatchability	

<i>C. dilutus</i>	<b>Du et al. 2013</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Control response 8	Not reported	
Temperature	23±1 °C	
Test type	Static renewal	150 mL twice daily
Photoperiod/light intensity	16 h/8 h dark	
Overlying water	Well water	
pH	7.51±0.16	
Hardness	Not Reported	<b>Doc. Points/ Accept. points</b>
Alkalinity	Not Reported	<b>Doc. Points/ Accept. points</b>
Conductivity	302±17 µmhos/cm	
Dissolved Oxygen	5.8±1.4 mg/L	
Ammonia-N	0.46±0.21 mg/L	
Sediment source	From drinking water reservoir in Conghua, China	
Organic carbon content	1.60±0.14%	
Particle size distribution (sand, silt, clay)	Sieved <500 µm	<b>Doc. points</b>
Percent moisture	49%	
Sediment spike method	Not reported	Mixed 4h, then again before bioassay initiation  <b>Doc. Points/ Accept. points</b>
Sediment spike equilibration time	17d @ 4°C	<b>Accept. points</b>

<i>C. dilutus</i>	<b>Du et al. 2013</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Sediment extraction/analysis method	Microwave extractor and GC/MS	
Interstitial water monitored?	Yes	
Interstitial water isolation method	Rolling incubator for 24h	
Interstitial water chemical extraction	Tenax	
Interstitial water chemical analysis	GC/MS	
DOC	Not reported	
Feeding	Yes	no feeding in the first 2 d, 0.6 g/L from days 3 to 7, 3 g/L from days 8 to 12, and then 6 g/L until the termination of bioassays
Purity of test substance	>97%	chemservice
Concentrations measured?	Yes	
Measured is what % of nominal?	Not reported	<b>Accept. points</b>
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Chemical method documented?	Yes	
Concentration of carrier (if any) in test solutions	<6uL acetone/g sediment	
Concentration 1 Meas (µg/g OC)	0.92±0.26	9 Reps and 20 per, nominal conc. not reported

<i>C. dilutus</i>	Du et al. 2013	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
		<b>Doc. Points</b>
Concentration 2 Meas (µg/g OC)	4.84±0.70	9 Reps and 20 per
Concentration 3 Meas (µg/ g OC)	6.08±1.76	9 Reps and 20 per
Concentration 4 Meas (µg/ g OC)	6.60±0.80	9 Reps and 20 per
Concentration 5 Meas (µg/ g OC)	9.84±1.41	9 Reps and 20 per
Concentration 6 Meas (µg/ g OC)	14.8±1.72	9 Reps and 20 per
Control	Solvent and negative controls	9 Reps and 20 per
LC <sub>5</sub> (µg/g OC)	20d: 0.143±0.022 58d: 0.075	58d estimated from Figure 5A
LC <sub>50</sub> (µg/g OC)	20d: 1.83±1.13 58d: 1.20±0.55	Method: Probit analyses with SPSS 13.0 software
EC <sub>5</sub> (µg/g OC)	Growth (20d): 0.034±0.006  Cumulative emergence: 0.016±0.008  Reproduction: 0.009±0.008	
EC <sub>50</sub> (µg/g OC)	Growth (20d): 1.09±0.56  Cumulative emergence: 0.838±0.77  Reproduction: 0.039±0.105	

Notes:

Protocol adapted from: USEPA, 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Protocol fulfills requirement of USEPA OPPTS 850.1735 Whole sediment acute toxicity invertebrates, freshwater (USEPA, 1996).

Reliability points taken off for:

Documentation: Nominal concentrations (2), Overlying water hardness (1), Overlying water alkalinity (1), Sediment particle size dist. (1), Sediment spike method (4), Hypothesis tests (8).

Total:  $100 - 17 = 83$

Acceptability: Measured concentration within 20% nominal (4), Spike method (4), Spike equilibration time (6), Carrier solvent evaporated (4), Organisms Randomly assigned (1), Overlying water Hardness (1), Overlying water Alkalinity (1), Random block design (2), Hypothesis tests (3). Total:  $100 - 26 = 74$

**Reliability Score: Mean (83, 74) = 78.5**

## Toxicity Data Summary

### *Chironomus dilutus*

Harwood AD, You J, Lydy MJ. (2009) Temperature as a toxicity identification evaluation tool for pyrethroid insecticides: Toxicokinetic confirmation. Environ. Toxicol. Chem. 28:1051-1058.

#### Relevance

Score: 100

Rating: R

#### Reliability

Score: 79.5

Rating: R

<i>C. dilutus</i>	<b>Harwood et al. 2009</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test method cited	USEPA 2000	600/R-99-064
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	3 <sup>rd</sup> instar larvae	
Source of organisms	Southern Illinois U. Lab culture	
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	
Animals randomized?	Not stated	<b>Accept. Points</b>
Test vessels randomized?	Not stated	<b>Accept. Points</b>



<i>C. dilutus</i>	Harwood et al. 2009	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	>85%	
Temperature	13 and 23°C	
Test type	Static	Daily renewal 75%
Photoperiod/light intensity	Not stated	<b>Doc. Points/ Accept. Points</b>
Overlying water source	Moderately hard water	
pH	6.7-7.2	
Hardness mg/L as CaCO <sub>3</sub>	Not stated	<b>Doc. Points/ Accept. Points</b>
Alkalinity mg/L as CaCO <sub>3</sub>	Not stated	<b>Doc. Points/ Accept. Points</b>
Conductivity	275-396 uS/cm	
Dissolved Oxygen	6.39-7.41 mg/L	
Sediment source	Touch of Nature reference site, Carbondale, IL	
Organic carbon content	0.69%	
Particle size distribution (sand, silt, clay)	Sieved <500 um	<b>Doc. points</b>
Sediment spike procedure	Dropwise acetone sol'n added to sediment slurry while mixing – mix 1 h	<b>Accept. points</b>
Sediment spike equilibration	14 d in dark -4°C	<b>Accept. points</b>

<i>C. dilutus</i>	Harwood et al. 2009	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
time		
Sediment to Solution ratio	0.60	
Sediment extraction/analysis method	Solvent extraction, cleanup, GC/ECD	
Interstitial water monitored?	No	
Interstitial water extraction method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
Interstitial water DOC	Not applicable	
Feeding	1 mL of 6 g/L of tetrafin sol'n daily	
Purity of test substance	>96%	
Concentrations measured?	Yes	
Measured is what % of nominal?	Not reported	<b>Accept. Points</b>
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Chemical method documented?	Yes	Solvent ext, cleanup, GC/ECD
Concentration of carrier (if any) in test solutions	Acetone	<b>Accept. points</b>
Concentration range Meas (ug/g OC)	4.26 to 76.9	Only conc. range given;  Nominal conc. not reported  <b>Doc. points</b>

<i>C. dilutus</i>	Harwood et al. 2009	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
		4 reps and 10 midges/rep
Controls	Solvent and negative	Pooled for analysis
LC50 (µg/g OC)	13C: 9.16 (3.48-18.5); 23C: 27.4 (19.0-37.8)	Log probit analysis

Notes:

Reliability points taken off for:

Documentation: Nominal concentrations (2), Overlying water hardness (1), Overlying water alkalinity (1), Photoperiod (1), Particle size distribution (1), Hypothesis tests (8). Total: 100-14=86

Acceptability: Measured Conc. within 20% of nominal (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent evaporation (4), Organisms randomly assigned (1), Overlying water hardness (1), Overlying water alkalinity (1), Photoperiod (1), Random block design (2), Hypothesis test (3). Total: 100-27=73

**Mean (86, 73) = 79.5**

## Toxicity Data Summary

*Chironomus dilutus* (formerly *C. tentans*)

Maul JD, Brennan AA, Harwood AD, Lydy MJ (2008a) Effect of sediment-associated pyrethroids, fipronil and metabolites on *Chironomus tentans* growth rate, body mass, condition index, immobilization and survival. Environ Toxicol Chem 27:2582-2590.

### Relevance

Score: 100 (Survival, Growth),  
85 (Immobility)  
Rating: R (Survival, Growth),  
L (Immobility)

### Reliability

Score: 82 (Survival, Growth),  
80.5 (Immobility)  
Rating: R

Relevance points taken off for: Immobility - Control response not reported (15).

<i>C. dilutus</i>	<b>Maul 2008</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test method cited	USEPA 2000	600/R-99-064
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	Formerly <i>C. tentans</i>
Family in North America?	Yes	
Age/size at start of test/growth phase	Mid to late 3 <sup>rd</sup> instar larvae	
Source of organisms	Lab culture	Southern Illinois U.
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	

<i>C. dilutus</i>	<b>Maul 2008</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Animals randomized?	Yes	
Test vessels randomized?	Yes	
Test duration	10 d	
Data for multiple times?	Not stated	
Effect 1	Survival	
Control response 1	84%-95% all compounds	
Effect 2	Immobilization	Defined as inability to perform typical S-shape response to probing
Control response 2	Not reported	<b>Accept. Points</b>
Effect 3	Growth - Body mass by ash free dry mass (AFDM) - Daily instantaneous growth rate (IGR)	
Control response 3	AFDM: 0.65 mg IGR: 1.007	Estimated from Fig. 1C AFDM response acceptable (>0.48 mg)
Effect 4	Body condition index (BCI)	Calculated by regressing AFDM of controls against head size score of exposed organisms
Control response 4	Not reported	
Temperature	23°C	
Test type	Static	Daily renewal 75%

<i>C. dilutus</i>	<b>Maul 2008</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Photoperiod/light intensity	16 h light:8 h dark	
Overlying water source	EPA reconstituted moderately hard water Moderately hard water	
pH	6.61-6.74	
Hardness mg/L as CaCO <sub>3</sub>	Not reported	<b>Doc./Accept. Points</b>
Alkalinity mg/L as CaCO <sub>3</sub>	Not reported	<b>Doc./Accept. points</b>
Conductivity	275-396 uS/cm	
Dissolved Oxygen	6.12-6.78 mg/L	
Sediment formulated?	No	Source: 15 km south of Carbondale, IL
Organic carbon	0.69%	
Particle size distribution (sand, silt, clay)	Not stated	Sieved to <500 µm
Sediment spike procedure	150 uL dropwise acetone sol'n added to sediment slurry while mixing – mix 1 h	<b>Accept. points</b>
Sediment spike equilibration time	14 d in dark 4°C	<b>Accept. points</b>
Sediment to Solution ratio	50 g dw: 700 mL	
Sediment extraction/analysis method	Solvent extraction, cleanup, GC/ECD	
Interstitial water monitored?	No	
Interstitial water extraction method	Not applicable	

<i>C. dilutus</i>	<b>Maul 2008</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
Interstitial water DOC	Not applicable	
Feeding	1 mL of 6 g/L of tetrafin sol'n daily	
Purity of test substance	>98%	Chem service
Concentrations measured?	Yes	
Measured is what % of nominal?	72.9 ± 6.0%	
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in test solutions	See spike procedure	
Concentration 1 Meas (µg/g OC)	4.3	5 reps/conc and 10 midges/rep Nominal conc. NR <b>Doc. points</b>
Concentration 2 Meas (µg/g OC)	8.5	5 reps/con and 10 midges/rep
Concentration 3 Meas (µg/g OC)	23.4	5 reps/con and 10 midges/rep
Concentration 4 Meas (µg/g OC)	38.2	5 reps/con and 10 midges/rep
Concentration 5 Meas (µg/g OC)	74.2	5 reps/con and 10 midges/rep
Concentration 6 Meas (µg/g OC)	77	5 reps/con and 10 midges/rep

<i>C. dilutus</i>	<b>Maul 2008</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Control	Solvent and negative	5 reps/con and 10 midges/rep
LC50 (95% confidence interval) µg/g OC	24.5 (5.7-58.9)	Method: Log probit analysis
EC50 (95% confidence interval) µg/g OC	Immobilization: 11.5 (7.8-15.4) Growth AFDM: 27.4 (14.4-60.9) Growth IGR: 27.2 (15.4-58.3)	Method: Maximum likelihood analysis
EC20 µg/g OC	Growth AFDM: 12.6 (0.0-31.1) Growth IGR: 13.7 (8.8-31.0)	Linear interpolation method
NOEC µg/g OC	AFDM: 38.2 IGR: 38.2	Linear interpolation method MSD: not reported <b>Doc./Accept. points</b>
LOEC µg/g OC	AFDM: 74.2 IGR: 74.2	
MATC (GeoMean NOEC,LOEC) (µg/kg)	AFDM: 53.2 IGR: 53.2	
% of control at NOEC	AFDM: 0.25/0.65*100=38.4% IGR: 1.003/1.007*100=99.6%	
% of control at LOEC	AFDM: 0.1/0.65*100=15.4% IGR: 1.000/1.007*100=99.3%	

Notes:

Article refers to NOEC values but not specifically stated.



Lethal to sublethal ratios were also reported:

LC50/EC50\_immobilization: 2.1

LC50/EC20\_AFDM: 1.9

LC50/EC20\_IGR: 1.8

Reliability points taken off for:

**Survival & Growth – point estimates: Mean (86, 78) = 82**

Documentation: Nominal concentrations (2), Overlying water hardness (1), Overlying water alkalinity (1), Particle size distribution (2), Hypothesis Testing (8). Total:  $100-14=86$

Acceptability: Sediment spike method (4), Spike equilibration time (6), Carrier solvent evaporation (4), Overlying water hardness (1), Overlying water alkalinity (1), Temperature variation NR (3), Hypothesis tests (3). Total:  $100-22=78$

**Immobility-point estimates and LOEC: Mean (92, 69) = 80.5**

Documentation: Nominal concentrations (2), Overlying water hardness (1), Overlying water alkalinity (1), Particle size distribution (2), Minimum significant difference (2). Total:  $100-8=92$

Acceptability: Control response within guideline (10), Sediment spike method (4), Spike equilibration time (6), Carrier solvent evaporation (4), Overlying water hardness (1), Overlying water alkalinity (1), Temperature variation NR (3), Minimum significant difference (1), NOEC compared to control (1). Total:  $100-31=69$

## Toxicity Data Summary

### *Chironomus dilutus*

Picard CR (2010b) 10-Day toxicity test exposing midges (*Chironomus dilutus*) to permethrin applied to formulated sediment under static-renewal conditions following OPPTS Draft Guideline 850.1735. Performed by Springborn Smithers Laboratories, Wareham, MA, Study No. 13656.6148; submitted to Pyrethroid Working Group, Washington, DC.

#### Relevance

Score: 100

Rating: R

#### Reliability

Score: 88.5

Rating: R

<i>C. dilutus</i>	<b>Picard 2010b</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test method cited	EPA 2000	
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	11 day old, 3 <sup>rd</sup> instar larvae	
Source of organisms	Lab culture	Environmental Consulting & Testing, Superior, WI
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	72 h

<i>C. dilutus</i>	<b>Picard 2010b</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Animals randomized?	Yes	
Test vessels randomized?	Not reported	<b>Accept. points</b>
Test duration	10 day	
Data for multiple times?	No	
Effect 1	Mortality	
Control response 1	Negative control: 94% Solvent control: 93%	Pooled control 93%
Effect 2	Growth (total dry wt. per organism)	
Control response 2	Negative control: 0.77 mg Solvent control: 0.79 mg	Min = 70% control surv. and 0.48 AFDW/midge
Temperature	22 -25 °C	<b>Accept. points</b>
Test type	Static renewal	50 mL/cycle;7 cycles per day
Photoperiod/light intensity	16 h/8 h dark; 510-960 lux	
Overlying water	Well water	
pH	6.9-7.4	
Hardness	72-80 mg/L as CaCO <sub>3</sub>	
Alkalinity	18-38 mg/L as CaCO <sub>3</sub>	
Conductivity	410-490 µmhos/cm	
Dissolved Oxygen	4.4 – 7.9 mg/L	<b>Accept. points</b>
Ammonia-N	0.54-2.2 @ d0 1.2 – 9.9 d10	

<i>C. dilutus</i>	<b>Picard 2010b</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Sediment source	Formulated	Method: OECD 218
Organic carbon	2.1%	
Particle size distribution (sand, silt, clay)	79%, 4%, 17%	
pH	6.8	
Percent solids	65.45%	
Sediment spike method	Jar rolling technique	4 h @ RT; 15 rpm
Sediment spike equilibration time	14 d @ 2 - 8°C	Mixed 2x/week for 2 h @ RT  <b>Accept. points</b>
Sediment to Solution ratio	100:175 mL	100 mL sediment = 156 g wet wt or 102 g dry wt
Sediment extraction/analysis method	Ext/cleanup and GC/MS	
Interstitial water monitored?	Yes	Results in supplemental report; not referenced
Interstitial water isolation method	Centrifugation	1200 g 15-30 min
Interstitial water chemical extraction	SPME	
Interstitial water chemical analysis	Not stated	
DOC	88-120 mg C/L	
Feeding	1.5 mL (4.0 mg/mL) flaked fish food susp.	Per vessel once daily

<i>C. dilutus</i>	<b>Picard 2010b</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Purity of test substance	95.1%	
Concentrations measured?	Yes	
Measured is what % of nominal?	79-95% in formulated sediment spikes	82-120% in stock solutions  <b>Accept. points</b>
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Chemical method documented?	Yes	Solvent Ext/ SPE cleanup and GCMS/NCI analysis
Concentration of carrier (if any) in test solutions	0%	10 mL of acetone evaporated from sand
Concentration 1 Nom; Meas (µg/kg)	63; 54	8 Reps and 10 per
Concentration 2 Nom; Meas (µg/kg)	130; 120	8 Reps and 10 per
Concentration 3 Nom; Meas (µg/kg)	250; 220	8 Reps and 10 per
Concentration 4 Nom; Meas (µg/kg)	500; 490	8 Reps and 10 per
Concentration 5 Nom; Meas (µg/kg)	1000; 880	8 Reps and 10 per
Concentration 6 Nom; Meas (µg/kg)	2000; 2000	8 Reps and 10 per
Control	Solvent and negative controls	8 Reps and 10 per
LC <sub>50</sub> (95% confidence interval)	Dry weight basis 430 (370-500) µg/kg DW  OC-normal basis 20.5 (17.6-23.8) µg/g OC	Method: Spontaneous Log – log analysis using TOXSTAT

<i>C. dilutus</i>	<b>Picard 2010b</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
EC <sub>50</sub> (95% confidence interval)	Dry weight basis 110 (90 – 130) µg/kg DW  OC-normal basis 5.2 (4.3-6.2) µg/g OC	Method: Linear interpretation method; empirically estimated
NOEC	Dry weight basis Survival: 120 µg/kg DW  Growth: < 54 µg/kg DW  OC-normal basis Survival: 5.7 µg/g OC Growth: <2.6 µg/g OC	Method: Not stated only TOXSTAT program  p: 0.05  MSD: Not reported <b>Doc./Accept. points</b>
LOEC	Dry weight basis Survival: 220 µg/kg DW  Growth: 54 µg/kg DW  OC-normal basis Survival: 10.5 µg/g OC Growth: 2.6 µg/g OC	Method: Not stated only TOXSTAT program  p: 0.05  MSD: Not reported
MATC (GeoMean NOEC,LOEC)	Dry weight basis Survival: 162 µg/kg DW  Growth: not able to calculate  OC-normal basis Survival: 7.7 µg/g OC Growth: not able to calculate	
% of control at NOEC	Survival: 95%/93%=102% Growth: not able to calculate	Pooled controls
% of control at LOEC	Survival: 50/93=54%  Growth: 0.61/0.78=78%	Pooled controls

Notes:

Protocol adapted from: USEPA, 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Protocol fulfills requirement of USEPA OPPTS 850.1735 Whole sediment acute toxicity invertebrates, freshwater (USEPA, 1996).

Measured sediment concentrations are the mean of measurements at day 0 and day 10.

Although the study states pore water results are in a supplemental report, the data was never made available due to analytical and sample holding time issues.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total:  $100-2=98$

Acceptability: Measured concentration within 20% nominal (4), Spike equilibration time (4), Dissolved oxygen < 60% saturation (5), Temperature variation (3), Random design (2), Minimum significant difference (1). Total:  $100-21=79$

**Reliability Score: Mean (98, 79) = 88.5**

## Toxicity Data Summary

### *Hyalella azteca*

Picard CR (2010a) 10-Day toxicity test exposing freshwater amphipods (*Hyalella azteca*) to permethrin applied to formulated sediment under static-renewal conditions. Performed by Springborn Smithers Laboratories, Wareham, MA, Study No. 13656.6138; submitted to Pyrethroid Working Group, Washington, DC.

#### Relevance

Score: 100

Rating: R

#### Reliability

Score: 95.5

Rating: R

<i>H. azteca</i>	<b>Picard 2010a</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyalellidae	
Genus	<i>Hyalella</i>	
Species	<i>azteca</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	8 day old	
Source of organisms	Springborn Smithers lab culture	
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	



<i>H. azteca</i>	Picard 2010a	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Animals randomized?	Yes	
Test vessels randomized?	Not reported	<b>Accept. points</b>
Test duration	10 day	
Effect 1	Survival	
Control response 1	97% neg control/95% solvent control survival	Pooled control
Effect 2	Growth	Dry weight
Control response 2	0.13 mg	Pooled control
Temperature	23±1 °C	
Test type	Static renewal	50 mL/cycle;7 cycles per day
Photoperiod/light intensity	16 h/8 h dark; 500-710 lux	
Overlying water	Well water	
pH	6.4-7.5	6.9-7.3 during test
Hardness	64-66 mg/L	64-72 during test
Alkalinity	19-21 mg/L	20-22 during test
Conductivity	410-450 µmhos/cm	380-400 during test
Dissolved Oxygen	6.3-8.0 mg/L during test	
TOC/DOC	0.49 mg/L/Not stated	
Ammonia-N	<0.01 – 0.49 mg/L during test	
Sediment source	Formulated	Method: OECD 218
Organic carbon	2.3%	
Particle size distribution (sand,	80%, 3%, 17%	

<i>H. azteca</i>	Picard 2010a	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
silt, clay)		
Sediment spike procedure	Jar rolling technique	4 h @ RT; 15 rpm
Sediment spike equilibration time	15 d @ 4°C	Mixed 2x/week for 2 h @ RT  <b>Accept. points</b>
Sediment to Solution ratio	100:175 mL	100 mL sediment = 147 g wet wt or 101 g dry wt
Interstitial water monitored?	Yes	Results in supplemental report; not referenced
Interstitial water isolation method	Centrifugation	1200 g 15-30 min
Interstitial water chemical extraction	SPME	
Interstitial water chemical analysis	Not stated	
Interstitial water DOC	120-150 mg C/L @0d	94-120 mg C/L @ 10d
Feeding	1 mL of YCT daily	Per replicate vessel
Purity of test substance	95.1%	
Concentrations measured?	Yes	
Measured is what % of nominal?	72-96% in sediment spikes	84-110% in stock solutions
Toxicity values calculated based on nominal or measured	Measured	

<i>H. azteca</i>	Picard 2010a	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
concentrations?		
Concentration of carrier (if any) in test solutions	0%	10 mL of acetone evaporated from sand
Concentration 1 Nom; Meas (µg/kg)	4.0; 3.4	8 Reps and 10 per
Concentration 2 Nom; Meas (µg/kg)	8.0; 7.4	8 Reps and 10 per
Concentration 3 Nom; Meas (µg/kg)	16; 13	8 Reps and 10 per
Concentration 4 Nom; Meas (µg/kg)	32; 26	8 Reps and 10 per
Concentration 5 Nom; Meas (µg/kg)	64; 53	8 Reps and 10 per
Concentration 6 Nom; Meas (µg/kg)	128; 100	8 Reps and 10 per
Control	Solvent and negative controls	8 Reps and 10 per
LC <sub>50</sub> (95% confidence interval)	Dry weight basis 60 (53-66) µg/kg DW  OC-normal basis 2.6 (2.3-2.9) µg/g OC	Method: Linear interpretation method using TOXSTAT
EC <sub>50</sub> (95% confidence interval)	Dry weight basis 46 (34-59) µg/kg DW  OC-normal basis 2.0 (1.5-2.6) µg/g OC	Method: Linear interpretation method; empirically estimated
NOEC	Dry weight basis Survival: 26 µg/kg DW  Growth: 7.4 µg/kg DW  OC-normal basis Survival: 1.1 µg/g OC Growth: 0.32 µg/g OC	Method: Not stated only TOXSTAT program  p: 0.05  MSD: not reported  <b>Doc./Accept.</b>

<i>H. azteca</i>	<b>Picard 2010a</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
		<b>points</b>
LOEC	Dry weight basis Survival: 53 µg/kg DW  Growth: 13 µg/kg DW  OC-normal basis Survival: 2.3 µg/g OC Growth: 0.57 µg/g OC	Same as above
MATC (GeoMean NOEC,LOEC)	Dry weight basis Survival: 37 µg/kg DW  Growth: 10 µg/kg DW  OC-normal basis Survival: 1.6 µg/g OC Growth: 0.42 µg/g OC	
% of control at NOEC	Survival: (93%/96%=97%); Growth: (0.12/0.13=92%)	Pooled controls
% of control at LOEC	Survival: (56/96=58%)  Growth: (0.11/0.13=85%)	Pooled controls

Notes:

Protocol adapted from: USEPA, 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Protocol fulfills requirement of USEPA OPPTS 850.1735 Whole sediment acute toxicity invertebrates, freshwater (USEPA, 1996).

Measured sediment concentrations are the mean of measurements at day 0 and day 10.

Although the study states pore water results are in a supplemental report, the data was never made available due to analytical and sample holding time issues.

Reliability points taken off for:

Documentation: Minimum significant difference (2). Total:  $100-2=98$

Acceptability: Spike equilibration time (4), Random design (2), Minimum significant difference (1). Total:  $100-7=93$

**Reliability Score: Mean (98, 93) = 95.5**

## Toxicity Data Summary

### *Hyaella azteca*

Weston DP, Jackson CJ (2009) Use of engineered enzymes to identify organophosphate and pyrethroid-related toxicity in toxicity identification evaluations. Environmental Science & Technology 43:5514-5520.

#### Relevance

Score: 100

Rating: R

#### Reliability

Score: 74

Rating: R

<i>H. azteca</i>	<b>Weston &amp; Jackson 2009</b>	
<b>Parameter</b>	<b>Value</b>	<b>Notes</b>
Results published or in signed, dated format?	Yes	
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyaellidae	
Genus	<i>Hyaella</i>	
Species	<i>azteca</i>	
Family relevant for North America?	Yes	
Age/size at start of test/growth phase	7-10 days	
Source of organisms	Lab culture	
Have organisms been pre-exposed to contaminants?	No	
Were animals acclimated and disease-free?	Yes	
Were animals randomized?	Not reported	<b>Accept. points</b>
Were test vessels randomized?	Not reported	<b>Accept. points</b>

<i>H. azteca</i>	<b>Weston &amp; Jackson 2009</b>	
<b>Parameter</b>	<b>Value</b>	<b>Notes</b>
Test duration	10 d	
Effect 1	Survival	
Control response 1	≥95%	
Temperature	23 ± 0.1°C	
Exposure type	Static renewal	100 mL water changed 3x/day
Photoperiod/light intensity	Not reported	<b>Doc. points</b>
Overlying water source	EPA moderately hard water	
pH	6.76 ± 0.13	
Hardness	Not reported	<b>Doc./Accept. points</b>
Alkalinity	Not reported	<b>Doc./Accept. points</b>
Dissolved oxygen	7.34 ± 0.32 mg/L	
Conductivity	367 ± 25 µS/cm	
Sediment source	3 natural soils/sediments: TON, BAY, LPH	
Organic carbon content	2.0%	
Particle size distribution (sand, silt, clay)	TON: 14%, 62%, 24% BAY: 52%, 38%, 10% LPH: 46%, 47%, 7%	
Sediment spike procedure	Sediment spiked in bulk in 1L jars with ≤200 µL acetone stock solutions; homogenized by hand mixing & rolling 1 h	Solvent not evaporated <b>Accept. points</b>
Sediment spike equilibration time	14 d at 4°C in darkness	<b>Accept. points</b>

<i>H. azteca</i>	<b>Weston &amp; Jackson 2009</b>	
<b>Parameter</b>	<b>Value</b>	<b>Notes</b>
Sediment-to-solution ratio	75 mL sediment:250 mL water	
Sediment extraction/analysis method	Extracted via Tenax for 6 h or 24 h, Tenax was solvent extracted & analyzed via LSC	
Interstitial water monitored?	Yes	
Interstitial water isolation method	Not applicable	
Interstitial water chemical analysis method	Extracted via SPME fibers equilibrated for 28 d on shaker table. Fibers extracted with hexane for 36 h; analyzed via LSC	
Interstitial water TOC; DOC	Not reported	
Feeding	1 mL of yeast-cerophyll-trout chow	
Purity of test chemical	<sup>14</sup> C-labeled: ≥ 98% Unlabeled: technical	
% Measured compared to nominal	Not reported	<b>Accept. points</b>
Were toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in test solutions	Acetone solvent not evaporated, conc. not reported	<b>Accept. points</b>
Concentration 1 Nom; Meas (mg/kg)	6-7 concentrations Actual concentrations not reported	4 reps, 10 per rep <b>Doc. points</b> - nom & meas concentrations not reported <b>Accept. points</b> – conc. spacing not reported



<i>H. azteca</i>	<b>Weston &amp; Jackson 2009</b>	
<b>Parameter</b>	<b>Value</b>	<b>Notes</b>
Control Type	Negative and solvent	4 reps, 10 per rep
Lab spike sediment LC <sub>50</sub> (95% confidence interval) ug/g OC	23.9 (23.4-24.4)	Method: probit
Agricultural-affected sediment LC <sub>50</sub> (95% confidence interval) ug/g OC	79.6 (63.1-100.3)	

Notes:

Reliability points taken off for:

Documentation: Nominal concentrations (2), Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Photoperiod (3), Hypothesis tests (8). Total: 100-25=75

Acceptability: Measured concentrations within 20% nominal (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organisms randomly assigned (1), Overlying water hardness (1), Overlying water alkalinity (1), Random design (2), Dilution factor (2), Hypothesis tests (3). Total: 100-28=72

**Reliability scores: Mean (75, 72)=73.5**

## ***Appendix 2***

Supplemental data rated RL, LR, or LL

## Toxicity Data Summary

### *Ampelisca abdita*

Anderson BD, Lowe S, Phillips BM, Hunt JW, Vorhees J, Clark S, Tjeerdema RS (2008)  
Relative sensitivities of toxicity test protocols with the amphipods *Eohaustorius estuarius* and *Ampelisca abdita*. *Ecotoxicology and Environmental Safety* 69:24-31.

#### Relevance

Score: 70

Rating: L

#### Reliability

Score: 63.5

Rating: L

\*Relevance points taken off for: saltwater species (15); control results not reported (15)

<i>A. abdita</i>	Anderson et al. 2008	
Parameter	Value	Comment
Test method cited	USEPA 1994	Estuarine and marine amphipods
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Ampeliscidae	
Genus	<i>Ampelisca</i>	
Species	<i>abdita</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	Not reported	<b>Doc./Accept. points</b>
Source of organisms	Lab culture	Brezina & Assoc.
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Not reported	<b>Accept. points</b>
Animals randomized?	Not reported	<b>Accept. points</b>
Test vessels randomized?	Not reported	<b>Accept. points</b>
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	Not reported	<b>Doc./Accept. points</b>
Temperature	20°C	
Test type	Static	
Photoperiod/light intensity	Not reported	<b>Doc. points</b>
Overlying water	Filtered seawater diluted with distilled water to 28 ‰ salinity	
pH	Not reported	<b>Accept. points</b>

<b><i>A. abdita</i></b>	<b>Anderson et al. 2008</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Hardness mg/L as CaCO <sub>3</sub>	Not reported	<b>Doc./Accept. points</b>
Alkalinity mg/L as CaCO <sub>3</sub>	Not reported	<b>Doc./Accept. points</b>
Conductivity	Not reported, 28 ‰ salinity	<b>Doc./Accept. points</b>
Dissolved Oxygen	Not measured. Slow aeration of test vessels	<b>Doc./Accept. points</b>
TOC/DOC	Not reported	
Chemical analysis?/Method	No	
Sediment formulated?	Yes	Equal parts Salinas River sediment from a reference site & clean sand, amended with 0.75% peat
Organic carbon	0.78%	
Particle size distribution (sand, silt, clay)	13.57% med sand; 48.17% fine sand; 38.27% silt+clay (% fines)	
pH	Not reported	<b>Doc. points</b>
Percent solids	350 mL water: 107.5 g sed	
Sediment spike procedure	50 mL acetone sol'n added to empty jar & allowed to evaporate; sediment & water added to jar & rolled for 1 <sup>st</sup> 24 h of eq. time	
Sediment spike equilibration time	7 d	<b>Accept. points</b>
Sediment to Solution ratio	50mL:200mL	
Interstitial water monitored?	No	
Interstitial water extraction method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
pH	Not applicable	
TOC; DOC	Not applicable	
Feeding	Not reported	<b>Doc./Accept. points</b>
Purity of test substance	Not reported	<b>Doc./Accept. points</b>
Concentrations measured?	Yes	
Measured is what % of nominal?	35.5-50.2%	<b>Accept. points</b>

<i>A. abdita</i>	Anderson et al. 2008	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Toxicity values calculated based on nominal or measured concentrations?	Nominal	
Chemical method documented?	Yes	EPA 1660
Concentration of carrier (if any) in test solutions	0 (evaporated)	
Concentration 1 Nom; Meas (mg/kg DW)	Test 1: 5.6; 2.809 Test 2: 5.6; 3.480	5 reps, 5 org/rep
Concentration 2 Nom; Meas (mg/kg DW)	Test 1: 10.0; 3.552 Test 2: 10.0; 5.856	5 reps, 5 org/rep
Concentration 3 Nom; Meas (mg/kg DW)	Test 1: 18.0; 7.655 Test 2: 18.0; 7.508	5 reps, 5 org/rep
Control	Solvent and negative	5 reps, 5 org/rep
LC <sub>50</sub>	Dry Weight Basis Test 1: 6.468 mg/kg DW Test 2: 10.358 mg/kg DW Mean: 8.913 (SD=7.463) mg/kg DW  OC-normal basis Test 1: 0.829 µg/g OC Test 2: 1.33 µg/g OC Mean: 1.14 (SD=0.96) µg/g OC	Method: ToxCalc software

Notes:

Reliability points taken off for:

Documentation: Organism age (5), Chemical purity (5), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Sediment pH (1), Photoperiod (3), Hypothesis tests (8). Total: 100-27=73

Acceptability: Control response (9), Chemical purity (10), Measured concentrations within 20% nominal (4), Sediment spike equilibration time (2), Organisms age (3), Organisms randomly assigned (1), Feeding (3), Organisms acclimated (1), Overlying water hardness (2), Overlying water alkalinity (2), Overlying water dissolved oxygen (6), Overlying water conductivity (1), Overlying water pH (2), Temperature variation (3), Random design (2), Hypothesis tests (3). Total: 100-54=46

**Reliability score: Mean (73, 46)=63.5**

## Toxicity Data Summary

### *Chironomus dilutes*

Ding Y, Landrum PF, You J, Lydy MJ (2013) Assessing bioavailability and toxicity of permethrin and DDT in sediment using matrix solid phase microextraction. *Ecotoxicology* 22:109–117

<u>Relevance</u>	<u>Reliability (Sediments 2 &amp; 3)</u>	<u>Reliability (Sediment 1)</u>
Score: 85	Score: 80.5	Score: 77.5
Rating: L	Rating: R	Rating: R

\*Relevance points taken off for: Acceptable control response not reported (15).

<i>C. dilutus</i>	<b>Ding 2013</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test method cited	EPA 2000	
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>dilutus</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	3 <sup>rd</sup> instar larvae	
Source of organisms	Lab culture	Fisheries and Illinois Aquaculture Center at Southern Illinois University
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	
Animals randomized?	Not reported	<b>Accept. Points</b>
Test vessels randomized?	Not reported	
Test duration	10 day	

<i>C. dilutus</i>	<b>Ding 2013</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Data for multiple times?	No	
Effect 1	Mortality	
Control response 1	Not Reported	<b>Relevance Points</b>
Temperature	23 °C	
Test type	Flow through	
Photoperiod/light intensity	16 h/8 h dark	
Overlying water	Not reported	<b>Doc./Accept. Points</b>
pH	7.6-7.9	
Hardness	Not reported	<b>Doc./Accept. Points</b>
Alkalinity	Not reported	<b>Doc./Accept. Points</b>
Conductivity	320-365 µmhos/cm	
Dissolved Oxygen	>6.0 mg/L	
Ammonia-N	<1.0 mg/L	
Sediment formulated?	No	
Organic carbon content	0.98 ± 0.10%	
Particle size distribution (sand, silt, clay)	14%, 72%, 14%	
pH	Not reported	
Percent moisture	Not reported	
Sediment spike method	vigorously mixed for 30 min. using motor driven paddle	<b>Accept. Points</b>
Sediment spike equilibration time	3 aging times: 7d, 28d and 90d at 23°C	Mixed initially and once more prior to use.
Sediment to Solution ratio	60g ww:250 mL	the dry wet ratio was approximately 20%
Sediment extraction/analysis method	Ext/cleanup and HPLC	
Interstitial water monitored?	Yes	

<i>C. dilutus</i>	<b>Ding 2013</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Interstitial water isolation method	matrix equilibration	vials of wet sediment, overlying water and HgCl <sub>2</sub> gently shaken for 28 days
Interstitial water chemical extraction	Matrix SPME	
Interstitial water chemical analysis	liquid scintillation counting (LSC)	
DOC	not reported	
Feeding	Tetrafin suspension daily	
Purity of test substance	98.0%	
Measured is what % of nominal?	Nominal is not reported	<b>Accept. points</b>
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in test solutions	Not reported	
Concentration 1 Meas (7 d; 28 d; 90 d aged sed) (µg/kg)	0.069; 0.068; 0.063	3 Reps each sed./10 organisms
Concentration 2 Meas (7 d; 28 d; 90 d aged sed) (µg/kg)	0.156; 0.134; 0.134	3 Reps each sed./10 organisms
Concentration 3 Meas (7 d; 28 d; 90 d aged sed) (µg/kg)	0.292; 0.297; 0.253	3 Reps each sed./10 organisms
Concentration 4 Meas (7 d; 28 d; 90 d aged sed) (µg/kg)	0.616; 0.603; 0.565	3 Reps each sed./10 organisms
Concentration 5 Meas (7 d; 28 d; 90 d aged sed) (µg/kg)	0.888; 0.827; 0.779	3 Reps each sed./10 organisms
Control	Solvent control with acetone	no other info reported on controls <b>Doc./Accept. points</b>
LC <sub>50</sub> (95% confidence interval) nmol/g dw	7d: 0.44 (0.23-0.62) 28d: 0.13 (0.04-0.23) 90d: 0.30 (0.25-0.36)  <u>Converted to ug/g OC</u> 7d: 17.6 ug/g OC 28d: 5.2 ug/g OC	Method: see notes



<i>C. dilutus</i>	<b>Ding 2013</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
	90d: 12.0 ug/g OC	
LC <sub>50</sub> (95% confidence interval) matrix SPME basis (normalized to PDMS conc.) nmol/mL PDMS	7d: 9.9 (2.6-14.5) 28d: 2.8 (1.0-4.8) 90d: 3.7 (3.1-4.4)	Method: see notes
NOEC	not reported	<b>Doc./Accept. points</b>
LOEC	not reported	<b>Doc./Accept. points</b>
MATC (GeoMean NOEC,LOEC)		
% of control at NOEC	Control response not reported	<b>Doc./Accept. points</b>
% of control at LOEC	Control response not reported	<b>Doc./Accept. points</b>

Notes:

Protocol adapted from: USEPA, 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Protocol fulfills requirement of USEPA OPPTS 850.1735 Whole sediment acute toxicity invertebrates, freshwater (USEPA, 1996).

Measured sediment concentrations are presented in the Supplemental Information, nominal concentrations were not reported:

**Table S1** Sediment concentrations of permethrin in *Chironomus dilutus* and *Hyalella azteca* tests. Data were presented as mean  $\pm$  standard deviation (n=3).

<i>Chironomus dilutus</i> test	7-d aging	28-d aging	90-d aging
concentration 1	0.069 $\pm$ 0.002	0.068 $\pm$ 0.003	0.063 $\pm$ 0.003
concentration 2	0.156 $\pm$ 0.014	0.134 $\pm$ 0.005	0.134 $\pm$ 0.002
concentration 3	0.292 $\pm$ 0.007	0.297 $\pm$ 0.006	0.253 $\pm$ 0.003
concentration 4	0.613 $\pm$ 0.018	0.606 $\pm$ 0.021	0.565 $\pm$ 0.010
concentration 5	0.888 $\pm$ 0.010	0.827 $\pm$ 0.056	0.779 $\pm$ 0.012
<i>Hyalella azteca</i> test	7-d aging	28-d aging	90-d aging
concentration 1	0.022 $\pm$ 0.001	0.021 $\pm$ 0.001	0.022 $\pm$ 0.001
concentration 2	0.040 $\pm$ 0.001	0.043 $\pm$ 0.001	0.047 $\pm$ 0.001
concentration 3	0.076 $\pm$ 0.001	0.067 $\pm$ 0.001	0.066 $\pm$ 0.001
concentration 4	0.143 $\pm$ 0.008	0.141 $\pm$ 0.002	0.151 $\pm$ 0.004
concentration 5	0.291 $\pm$ 0.008	0.279 $\pm$ 0.013	0.284 $\pm$ 0.006

Statistical analysis methods not clearly reported:

“Median lethal sediment concentration (LC50) (nmol/g dw) for permethrin, median lethal tissue residue (LR50) (nmol/g ww lipid), and median lethal fiber concentration (LC50 fiber) (nmol/mL PDMS) estimated by either using standard log-probit analysis (SAS Version 8.02, SAS Institute Cary, NC, USA) or trimmed Spearman-Kärber analyses (TSK), and Abbott’s correction (Abbott 1925) was applied in cases where the data were non-monotonic. Values with overlapping confidence intervals were considered to not be significantly different.”

Reliability points taken off for:

Documentation: Nominal concentrations in interstitial water and/or sediment (2); Overlying water: Source (2), Hardness (1), Alkalinity (1); Significance level (2); % of control at NOEC and/or LOEC (2).

Total:  $100 - 10 = 90$

Acceptability: Control response was within test guidance (10), Measured concentration within 20% nominal (4), \*Spike equilibration time (6), Carrier solvent fully evaporated (4), Organisms randomly assigned to test containers (1), Overlying water source acceptable (2), Hardness within organism tolerance (1), Alkalinity within organism tolerance (1), Random design (2), Adequate replication (2), NOEC response compared to control (1), LOEC response compared to control (1).

\*This is for Sediment 1 (aged 7 d) only.

Total (Sed 2 & 3):  $100 - 29 = 71$

Total (Sed 1)\*:  $100 - 35 = 65$

**Reliability Score: Mean (90, 69) = 80.5 for Sediments 2 & 3 (aged 28 & 90 d, respectively)**

**\* Reliability Score: Mean (90, 63) = 77.5 for Sediment 1 (aged 7 d)**

## Toxicity Data Summary

### *Chironomus riparius*

Conrad AU, Fleming RJ, Crane M (1999) Laboratory and field response of *Chironomus riparius* to a pyrethroid insecticide. Wat. Res. 33:1603-1610.

Laboratory SSTT:

Relevance

Score: 70

Rating: L

Reliability

Score: 65

Rating: L

\*Reasons for less than 100 pts for relevance: Chemical purity not stated (15), Toxicity values not based on measured sediment concentration (15)

Outdoor Pond

Reliability

Score: 63

Rating: L

<i>C. riparius</i>	<b>Conrad 1999</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test method cited	OECD 1995	
Phylum	Arthropoda	
Class	Insecta	
Order	Diptera	
Family	Chironomidae	
Genus	<i>Chironomus</i>	
Species	<i>riparius</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	Larvae hatched w/in 48 h allowed to mature for 8 d before test	

<i>C. riparius</i>	<b>Conrad 1999</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Source of organisms	Not stated	<b>Doc. Points</b>
Have organisms been exposed to contaminants?	Not stated	<b>Accept. Points</b>
Animals acclimated and disease-free?	Not stated	
Animals randomized?	Yes	
Test vessels randomized?	Not stated	
Test duration	10 d	
Data for multiple times?	Not stated	
Effect 1	Survival	
Control response 1	>90%	
Temperature	20°C	
Test type	Static	
Photoperiod/light intensity	16 h: 8h dark	
Overlying water source	Dechlorinated water	Royal Holloway groundwater
pH	Not stated	<b>Doc. Points/ Accept. Points</b>
Hardness mg/L as CaCO <sub>3</sub>	Not stated	<b>Doc. Points/ Accept. Points</b>
Alkalinity mg/L as CaCO <sub>3</sub>	Not stated	<b>Doc. Points/ Accept. Points</b>
Conductivity	Not stated	<b>Doc. Points/ Accept. Points</b>
Dissolved Oxygen	Not stated	<b>Doc. Points/ Accept. Points</b>

<i>C. riparius</i>	Conrad 1999	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Sediment formulated?	No	collected from an uncontaminated experimental pond, Medmenham UK
Organic carbon	9.64%	
Particle size distribution (sand, silt, clay)	sieved to 500 mm	
pH	Not stated	
Percent solids	51%	
Sediment spike procedure	Add acetone sol'n dropwise to 300 mL wet sediment while stirring; mix 1 h	<b>Accept. Points</b>
Sediment spike equilibration time	24 h	
Sediment to Solution ratio	50 mL:200 mL	
Sediment extraction/analysis method		
Interstitial water monitored?	Predicted	
Interstitial water extraction method	Not stated	
Interstitial water chemical extraction method	Not stated	
Interstitial water chemical analysis method	Not stated	
Interstitial water DOC	Not stated	
Feeding	None	
Purity of test substance	Not stated	
Concentrations measured?	No	

<i>C. riparius</i>	<b>Conrad 1999</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Measured is what % of nominal?	Not measured in lab SSTT	
Toxicity values calculated based on nominal or measured concentrations?	Nominal	<b>Rel. Points/ Doc. Points/ Accept. Points</b>
Chemical method documented?	No	
Concentration of carrier (if any) in test solutions	Not stated	
Concentration 1 Nom/Meas (ng/g dw)	0.43/ Not stated	3 rep./ 15 per <b>Accept. Points</b>
Concentration 2 Nom/Meas (ng/g dw)	4.3/ Not stated	3 rep./ 15 per
Concentration 3 Nom/Meas (ng/g dw)	22/ Not stated	3 rep./ 15 per
Concentration 4 Nom/Meas (ng/g dw)	43/ Not stated	3 rep./ 15 per
Concentration 5 Nom/Meas (ng/g dw)	220/ Not stated	3 rep./ 15 per
Concentration 6 Nom/Meas (ng/g dw)	430/ Not stated	3 rep./ 15 per
Control	Negative control	3 rep./ 15 per
LC50 (µg/g)	Dry weight basis 2.11 µg/g (1.83-2.40 µg/g) OC Basis (calculated) 21.9 µg/g OC	
EC50 (µg/kg)	Not stated	
NOEC (µg/kg)	Not stated	
LOEC (µg/kg)	Not stated	
MATC (GeoMean NOEC,LOEC) (µg/kg)	Not stated	
% of control at NOEC	Not stated	

<i>C. riparius</i>	Conrad 1999	
Parameter	Value	Comment
% of control at LOEC	Not stated	

Notes:

Reliability points taken off for:

Documentation: Organism source (2), Chemical purity (5), Measured concentrations (10), Overlying water hardness (1), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water conductivity (1), Overlying water pH (1), Hypothesis tests (8). Total: 100-30=70

Acceptability: Chemical purity (10), Measured concentrations (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent evaporation (4), Organisms acclimated (1), Overlying water hardness (1), Overlying water alkalinity (1), Conductivity (1), pH (1), random block (2), Adequate replication (2), Hypothesis tests (3). Total: 100-40=60

**Mean (70, 60) = 65**

Outdoor pond study:

Notes:

Sediment and porewater concentrations were estimated based on nominal water concentrations in the outdoor pond study using a Koc 24547 and OM%9.64.

Pond recovery determined by emergence in the highest dose pond was within 4 weeks and was similar to control after 2 months.

Documentation & Acceptability (Table 11) points taken off for:

Adequate range of organisms in system (6), Hardness reported (1), Alkalinity reported (1), Conductivity reported (1), Photoperiod reported (1), Organic carbon reported (2), Chemical fate reported (3), Species abundance reported (3), Species diversity reported (3), Biomass reported (2), Ecosystem recovery reported (2), Dose-response relationship observed (2), NOEC determined (4), Significance level stated (2), Minimum significant difference reported (2), % of control at NOEC and/or LOEC reported or calculable (2)

**Total: 100-37=63**

## Toxicity Data Summary

### *Eohaustorius estuarius*

Anderson BS, Lowe S, Phillips BM, Hunt JW, Vorhees J, Clark S, Tjeerdema RS (2008)  
Relative sensitivities of toxicity test protocols with the amphipods *Eohaustorius estuarius* and *Ampelisca abdita*. *Ecotoxicology and Environmental Safety* 69:24-31.

#### Relevance

Score: 70

Rating: L

#### Reliability

Score: 63.5

Rating: L

\*Relevance points taken off for: saltwater species (15); control results not reported (15)

<i>E. estuarius</i>	Anderson et al. 2008	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test method cited	USEPA 1994	Estuarine and marine amphipods
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Haustoriidae	
Genus	<i>Eohaustorius</i>	
Species	<i>estuarius</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	Not reported	<b>Doc./Accept. points</b>
Source of organisms	Lab culture	Northwestern Aquatic Sciences
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Not reported	<b>Accept. points</b>
Animals randomized?	Not reported	<b>Accept. points</b>
Test vessels randomized?	Not reported	<b>Accept. points</b>
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	



<i>E. estuarius</i>	Anderson et al. 2008	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Control response 1	Not reported	<b>Doc./Accept. points</b>
Temperature	15°C	
Test type	Static	
Photoperiod/light intensity	Not reported	<b>Doc. points</b>
Overlying water	Filtered seawater diluted with distilled water to 20 ‰ salinity	
pH	Not reported	<b>Accept. points</b>
Hardness mg/L as CaCO <sub>3</sub>	Not reported	<b>Doc./Accept. points</b>
Alkalinity mg/L as CaCO <sub>3</sub>	Not reported	<b>Doc./Accept. points</b>
Conductivity	Not reported, 20 ‰ salinity	<b>Doc./Accept. points</b>
Dissolved Oxygen	Not measured. Slow aeration of test vessels	<b>Doc./Accept. points</b>
TOC/DOC	Not reported	
Chemical analysis?/Method	No	
Sediment formulated?	Yes	Equal parts Salinas River sediment from a reference site & clean sand, amended with 0.75% peat
Organic carbon	0.78%	
Particle size distribution (sand, silt, clay)	13.57% med sand; 48.17% fine sand; 38.27% silt+clay (% fines)	
pH	Not reported	<b>Doc. points</b>
Percent solids	350 mL water: 107.5 g sed	
Sediment spike procedure	50 mL acetone sol'n added to empty jar & allowed to evaporate; sediment & water added to jar & rolled for 1 <sup>st</sup> 24 h of eq. time	
Sediment spike equilibration time	7 d	<b>Accept. points</b>

<i>E. estuarius</i>	Anderson et al. 2008	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Sediment to Solution ratio	50mL:200mL	
Interstitial water monitored?	No	
Interstitial water extraction method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
pH	Not applicable	
TOC; DOC	Not applicable	
Feeding	Not reported	<b>Doc./Accept. points</b>
Purity of test substance	Not reported	<b>Doc./Accept. points</b>
Concentrations measured?	Yes	
Measured is what % of nominal?	48.4-70.0%	<b>Accept. points</b>
Toxicity values calculated based on nominal or measured concentrations?	Nominal	
Chemical method documented?	Yes	EPA 1660
Concentration of carrier (if any) in test solutions	0 (evaporated)	
Concentration 1 Nom; Meas (mg/kg DW)	Test 1: 0.056; 0.035 Test 2: 0.056; 0.037	5 reps, 5 org/rep
Concentration 2 Nom; Meas (mg/kg DW)	Test 1: 0.100; 0.070 Test 2: 0.100; 0.0592	5 reps, 5 org/rep
Concentration 3 Nom; Meas (mg/kg DW)	Test 1: 0.560; 0.271 Test 2: 0.560; 0.374	5 reps, 5 org/rep
Control	Solvent and negative	5 reps, 5 org/rep

<i>E. estuarius</i>	Anderson et al. 2008	
Parameter	Value	Comment
LC <sub>50</sub>	Dry Weight Basis Test 1: 0.143 mg/kg DW Test 2: 0.147 mg/kg DW Mean: 0.140 (SD=0.143) mg/kg DW  OC-normal basis Test 1: 18.3 µg/g OC Test 2: 18.8 µg/g OC Mean: 17.9 (SD=1.88) µg/g OC	Method: ToxCalc software

Notes:

Reliability points taken off for:

Documentation: Organism age (5), Chemical purity (5), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Sediment pH (1), Photoperiod (3), Hypothesis tests (8). Total: 100-27=73

Acceptability: Control response (9), Chemical purity (10), Measured concentrations within 20% nominal (4), Sediment spike equilibration time (2), Organisms age (3), Organisms randomly assigned (1), Feeding (3), Organisms acclimated (1), Overlying water hardness (2), Overlying water alkalinity (2), Overlying water dissolved oxygen (6), Overlying water conductivity (1), Overlying water pH (2), Temperature variation (3), Random design (2), Hypothesis tests (3). Total: 100-54=46

**Reliability score: Mean (73, 46)=63.5**

## Toxicity Data Summary

### *Hyalella azteca*

Amweg EL, Weston DP, Ureda NM (2005) Use and toxicity of pyrethroid pesticides in the Central Valley, California, UAS. Environ Toxicol Chem 24: 966-972.

#### Relevance

Score: 85

Rating: L

#### Reliability

Score: 70

Rating: L

\*Relevance points taken off for: Toxicity values were not based on acceptable bioavailable concentrations (15). They were based on nominal (not measured) concentrations.

<b><i>H. azteca</i></b>	<b>Amweg et al. 2005</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyalellidae	
Genus	<i>Hyalella</i>	
Species	<i>azteca</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	6-10 d	< 350 µm, < 500 µm
Source of organisms	Not reported	<b>Doc. points</b>
Have organisms been exposed to contaminants?	Not reported	<b>Accept. points</b>
Animals acclimated and disease-free?	Not reported	<b>Accept. Points</b>
Animals randomized?	Not reported	<b>Accept. Points</b>
Test vessels randomized?	Not reported	<b>Accept. Points</b>
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	94%	
Effect 2	Growth	

<i>H. azteca</i>	<b>Amweg et al. 2005</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Control response 2	Negative: 78-85 µg Solvent: 76-90 µg	From Fig. 2E
Temperature	23°C	<b>Accept. Points</b>
Test type	Static-renewal	80% renewal every other day
Photoperiod/light intensity	16:8 h/ not stated	
Dilution water (overlying water)	Moderately hard water	Reconstituted from MQ water
pH	Measured, Not reported	<b>Doc. points</b>
Hardness mg/L as CaCO <sub>3</sub>	Measured, Not reported	<b>Doc. points</b>
Alkalinity mg/L as CaCO <sub>3</sub>	Measured, Not reported	<b>Doc. points</b>
Conductivity	Measured, Not reported	<b>Doc. points</b>
Dissolved Oxygen	Measured, Not reported	<b>Doc. points</b>
Sediment source	3 Natural sediments: American River (AR) Del Puerto Creek (DPC) Pacheco Creek (PC)	
Organic carbon content	AR: 1.4% DPC: 1.1% PC: 6.5%	
Particle size distribution (sand, silt, clay)	% silts & clays AR: 43.1% DPC: 31.7% PC: 21.3%	
Sediment spike procedure	<200 µL acetone /kg , mixed with electric drill	<b>Accept. points</b>
Sediment spike equilibration time	11-12 day at 4°C	<b>Accept. points</b>
Sediment to Solution ratio	50-75 mL:300 mL water	
Sediment extraction/analysis method	Solvent extraction, cleanup, GC/ECD	
Interstitial water monitored?	No	
Interstitial water isolation method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	

<i>H. azteca</i>	Amweg et al. 2005	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
DOC	Not applicable	
Feeding	Yeast, cerophyll, trout chow mix	Daily; no amounts
Purity of test substance	> 98%	Chem service
Concentrations measured?	Yes	
Measured is what % of nominal?	67%	<b>Accept. points</b>
Toxicity values calculated based on nominal or measured concentrations?	Nominal	
Concentration of carrier (if any) in test solutions	<200 µL acetone/kg wet sediment	<b>Accept. Points</b>
Concentration 1 Nom (µg/g OC)	0.057	10 per rep; 3 reps -Meas. conc. NR <b>Doc./Accept. points</b>
Concentration 2 Nom (µg/g OC)	0.09	10 per rep; 3 reps
Concentration 3 Nom (µg/g OC)	0.1	10 per rep; 3 reps
Concentration 4 Nom (µg/g OC)	0.2	10 per rep; 3 reps
Concentration 5 Nom (µg/g OC)	0.3	10 per rep; 3 reps
Concentration 6 Nom (µg/g OC)	0.6	10 per rep; 3 reps
Concentration 7 Nom (µg/g OC)	0.9	10 per rep; 3 reps
Concentration 8 Nom (µg/g OC)	1.6	10 per rep; 3 reps
Control	Solvent and negative	10 per rep; 3 reps
OC-normalized LC <sub>50</sub> (95% confidence interval) µg/g OC	AR: 17.87 (14.7-19.8) DPC: 11.10 (9.68-12.3) PC: 3.51 (2.86-4.18)	Method: trimmed Spearman-Kärber
Dry weight based LC <sub>50</sub> (95% confidence interval) ng/g DW	AR: 249 (206–277) DPC: 127 (111–142) PC: 226 (189–271)	Method: trimmed Spearman-Kärber
NOEC (µg/g OC)	Growth (from Fig. 2A) AR: 6.99 DPC: 6.99 PC: <1.51	Method: one-tailed Bonferroni's t-test p: 0.05 MSD: not reported <b>Doc./Accept. points</b>
LOEC (µg/g OC)	Growth AR: 11.77 DPC: 11.77 PC: 1.51	Method: one-tailed Bonferroni's t-test p: 0.05 MSD: not reported

<i>H. azteca</i>	Amweg et al. 2005	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
		<b>Doc./Accept. points</b>
MATC (GeoMean NOEC,LOEC) (µg/kg)	Growth AR: 9.07 DPC: 9.07 PC: can't calculate	Calculated
% of control at NOEC	Growth AR: 58/80*100=73% DPC: 68/78*100=87% PC: can't calculate	Estimated from Fig. 2E with solvent control results
% of control at LOEC	Growth AR: 50/80*100=63% DPC: 41/78*100=53% PC: 50/92*100=54%	Estimated from Fig. 2E with solvent control results

Notes:

Protocol follows EPA 2000 "Methods for measuring the toxicity and bioaccumulation. 2<sup>nd</sup>. EPA/600/R-99/064.

The above LC<sub>50</sub> values and test concentrations have been corrected as directed in the erratum to the original article. Permethrin correction factor was 2.22.

Reliability points taken off for:

Documentation: Organism source (4), Measured concentrations (10), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Overlying water pH (1), Minimum significant difference (2). Total: 100-22=78

Acceptability: Measured concentration within 20% (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organism not contaminated prior (3), Organisms randomly assigned (1), Organisms properly acclimated (1), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (5), Overlying water conductivity (1), Overlying water pH (1), Temperature variation (3), Random design (2), Minimum significant difference (1). Total: 100-38=62

**Reliability score: Mean (78, 62) = 70**

## Toxicity Data Summary

### *Hyalella azteca*

Amweg EL, Weston DP, Johnson CS, You J, Lydy MJ (2006) Effect of piperonyl butoxide on permethrin toxicity in the amphipon *Hyalella azteca*. Environ Toxicol Chem 25(7): 1817–1825.

#### Relevance

Score: 90

Rating: R

#### Reliability

Score: 73

Rating: L

\*Relevance points taken off for: Acceptable method used (10)

<i>H. azteca</i>	Amweg et al. 2006	
Parameter	Value	Comment
Test method cited	Not reported	Rel. points
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyalellidae	
Genus	<i>Hyalella</i>	
Species	<i>azteca</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	7-10 d	< 350 µm, < 500 µm
Source of organisms	Not reported	Doc. points
Have organisms been exposed to contaminants?	Not reported	Accept. points
Animals acclimated and disease-free?	Not reported	Accept. Points
Animals randomized?	Not reported	Accept. Points
Test vessels randomized?	Not reported	Accept. Points
Test duration	10 d	
Data for multiple times?	No	
Effect 1	Survival	
Control response 1	96±4%	



<i>H. azteca</i>	Amweg et al. 2006	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Temperature	23°C	<b>Accept. Points</b>
Test type	Static-renewal	80% renewal every other day
Photoperiod/light intensity	16:8 h/ not stated	
Overlying water source	Moderately hard water	Reconstituted from MQ water
pH	Measured, Not reported	<b>Doc. points</b>
Hardness mg/L as CaCO <sub>3</sub>	Measured, Not reported	<b>Doc. points</b>
Alkalinity mg/L as CaCO <sub>3</sub>	Measured, Not reported	<b>Doc. points</b>
Conductivity	Measured, Not reported	<b>Doc. points</b>
Dissolved Oxygen	Measured, Not reported	<b>Doc. points</b>
Sediment source	Natural sediment, S. Fork of American River, CA	
Organic carbon content	1.87%	
Particle size distribution (sand, silt, clay)	Not reported,	
Sediment spike procedure	<200 µL acetone /kg , mixed with electric drill	<b>Accept. points</b>
Sediment spike equilibration time	11-12 day at 4°C	<b>Accept. points</b>
Sediment to Solution ratio	50-75 mL:300 mL water	
Sediment extraction/analysis method	Solvent extraction, cleanup, GC/ECD	
Interstitial water monitored?	No	
Interstitial water isolation method	Not applicable	
Interstitial water chemical extraction method	Not applicable	
Interstitial water chemical analysis method	Not applicable	
DOC	Not applicable	
Feeding	Yeast, cerophyll, trout chow mix	Daily; no amounts
Purity of test substance	> 98%	Chem service
Concentrations measured?	Yes	

<i>H. azteca</i>	Amweg et al. 2006	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Measured is what % of nominal?	61.8 to 77.1%	<b>Accept. points</b>
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in test solutions	<200 µL acetone/kg wet sediment	<b>Accept. Points</b>
Concentration 1 Nom; Meas (µg/kg)	352.0; 217.7	10 per rep; 2 reps <b>Accept. points</b>
Concentration 2 Nom; Meas (µg/kg)	235.0; 144.1	10 per rep; 2 reps
Concentration 3 Nom; Meas (µg/kg)	157.0; 110.7	10 per rep; 2 reps
Concentration 4 Nom; Meas (µg/kg)	157.0; 117.1	10 per rep; 2 reps
Concentration 5 Nom; Meas (µg/kg)	104.0; 80.2	10 per rep; 2 reps
Control	Solvent	10 per rep; 2 reps
LC <sub>50</sub> (95% confidence interval) µg/g OC	(1) 14.2 (11.8-17.1) (2) 21.3 (14.7-30.5) (control) 13.2 (11.5-15.2)	Method: trimmed Spearman-Kärber, No sig diff. b/t permethrin LC50 and control LC50
NOEC (µg/g OC)		MSD: not reported <b>Doc./Accept. points</b>
LOEC (µg/g OC)		MSD: not reported <b>Doc./Accept. points</b>
MATC (GeoMean NOEC,LOEC) (µg/kg)		
% of control at NOEC		
% of control at LOEC		

Notes:

Reliability points taken off for:

Documentation : Organism source (4), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (2), Overlying water conductivity (1), Overlying water pH (1), Minimum significant difference (2). Total: 100-12=88

Acceptability : Measured concentration within 20% (4), Sediment spike method (4), Spike equilibration time (6), Carrier solvent (4), Organism not contaminated prior (3), Organisms randomly assigned (1), Organisms properly acclimated (1), Overlying water hardness (1), Overlying water alkalinity (1), Overlying water dissolved oxygen (5), Overlying water conductivity (1), Overlying water pH (1), Temperature variation (3), Random design (2), Replication (2), Hypothesis tests (3). Total: 100-42=58

**Reliability score: Mean (88, 58) = 73**

## Toxicity Data Summary

### *Hyalella azteca*

Ding Y, Landrum PF, You J, Lydy MJ (2013) Assessing bioavailability and toxicity of permethrin and DDT in sediment using matrix solid phase microextraction. *Ecotoxicology* 22:109–117

<u>Relevance</u>	<u>Reliability (Sediments 2 &amp; 3)</u>	<u>Reliability (Sediment 1)</u>
Score: 85	Score: 80.5	Score: 77.5
Rating: L	Rating: R	Rating: R

\*Relevance points taken off for: Acceptable control response not reported (15).

<b><i>H. azteca</i></b>	<b>Ding et al. 2013</b>	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Test method cited	EPA 2000	
Phylum	Arthropoda: Crustacea	
Class	Malacostraca	
Order	Amphipoda	
Family	Hyalellidae	
Genus	<i>Hyalella</i>	
Species	<i>azteca</i>	
Family in North America?	Yes	
Age/size at start of test/growth phase	1-2 weeks old	
Source of organisms	Lab culture	Fisheries and Illinois Aquaculture Center at Southern Illinois University
Have organisms been exposed to contaminants?	No	
Animals acclimated and disease-free?	Yes	
Animals randomized?	Not reported	<b>Accept. Points</b>
Test vessels randomized?	Not reported	
Test duration	10 day	

<i>H. azteca</i>	Ding et al. 2013	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Data for multiple times?	No	
Effect 1	Mortality	
Control response 1	Not Reported	<b>Relevance Points</b>
Temperature	23 °C	
Test type	Flow through	
Photoperiod/light intensity	16 h/8 h dark	
Overlying water	Not reported	<b>Doc./Accept. Points</b>
pH	7.6-7.9	
Hardness	Not reported	<b>Doc./Accept. Points</b>
Alkalinity	Not reported	<b>Doc./Accept. Points</b>
Conductivity	320-365 µmhos/cm	
Dissolved Oxygen	>6.0 mg/L	
Ammonia-N	<1.0 mg/L	
Sediment formulated?	No	
Organic carbon content	0.98 ± 0.10%	
Particle size distribution (sand, silt, clay)	14%, 72%, 14%	
pH	Not reported	
Percent moisture	Not reported	
Sediment spike method	vigorously mixed for 30 min. using motor driven paddle	<b>Accept. Points</b>
Sediment spike equilibration time	3 aging times: 7, 28 and 90d at 23°C	Mixed initially and once more prior to use.
Sediment to Solution ratio	60g ww:250 mL	the dry wet ratio was approximately 20%
Sediment extraction/analysis method	Ext/cleanup and HPLC	
Interstitial water monitored?	Yes	

<i>H. azteca</i>	Ding et al. 2013	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
Interstitial water isolation method	matrix equilibration	vials of wet sediment, overlying water and HgCl <sub>2</sub> gently shaken for 28 days
Interstitial water chemical extraction	Matrix SPME	
Interstitial water chemical analysis	liquid scintillation counting (LSC)	
DOC	not reported	
Feeding	Yeast-cerophyl-trout chow daily	
Purity of test substance	98.0%	
Measured is what % of nominal?	Nominal is not reported	<b>Accept. points</b>
Toxicity values calculated based on nominal or measured concentrations?	Measured	
Concentration of carrier (if any) in test solutions	Not reported	
Concentration 1 Meas (7 d; 28 d; 90 d aged sed) (µg/kg)	0.022; 0.021; 0.022	3 Reps each sed./10 organisms
Concentration 2 Meas (7 d; 28 d; 90 d aged sed) (µg/kg)	0.040; 0.043; 0.047	3 Reps each sed./10 organisms
Concentration 3 Meas (7 d; 28 d; 90 d aged sed) (µg/kg)	0.076; 0.067; 0.066	3 Reps each sed./10 organisms
Concentration 4 Meas (7 d; 28 d; 90 d aged sed) (µg/kg)	0.143; 0.141; 0.151	3 Reps each sed./10 organisms
Concentration 5 Meas (7 d; 28 d; 90 d aged sed) (µg/kg)	0.291; 0.279; 0.284	3 Reps each sed./10 organisms
Control	Solvent control with acetone	no other info reported on controls <b>Doc./Accept. points</b>
LC <sub>50</sub> (95% confidence interval) nmol/g dw	7d: 0.19 (0.17-0.21) nmol/g dw 28d: 0.16 (0.10-0.23) nmol/g dw 90d: 0.16 (0.10-0.26) nmol/g dw	Method: see notes

<i>H. azteca</i>	Ding et al. 2013	
<b>Parameter</b>	<b>Value</b>	<b>Comment</b>
	OC normalized (calculated) 7d: 7.59 ug/g OC 28d: 6.39 ug/g OC 90d: 6.39 ug/g OC	
LC <sub>50</sub> (95% confidence interval) matrix SPME basis (normalized to PDMS conc.) nmol/mL PDMS	7d: 4.5 (4.0-4.9) 28d: 3.7 (2.5-4.8) 90d: 2.2 (1.0-4.2)	Method: see notes
NOEC	not reported	<b>Doc./Accept. points</b>
LOEC	not reported	<b>Doc./Accept. points</b>
MATC (GeoMean NOEC,LOEC)		
% of control at NOEC	Control response not reported	<b>Doc./Accept. points</b>
% of control at LOEC	Control response not reported	<b>Doc./Accept. points</b>

Notes:

Protocol adapted from: USEPA, 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Protocol fulfills requirement of USEPA OPPTS 850.1735 Whole sediment acute toxicity invertebrates, freshwater (USEPA, 1996).

Measured sediment concentrations are presented in the Supplemental Information, nominal concentrations were not reported:

**Table S1** Sediment concentrations of permethrin in *Chironomus dilutus* and *Hyalella azteca* tests. Data were presented as mean  $\pm$  standard deviation (n=3).

<i>Chironomus dilutus</i> test	7-d aging	28-d aging	90-d aging
concentration 1	0.069 $\pm$ 0.002	0.068 $\pm$ 0.003	0.063 $\pm$ 0.003
concentration 2	0.156 $\pm$ 0.014	0.134 $\pm$ 0.005	0.134 $\pm$ 0.002
concentration 3	0.292 $\pm$ 0.007	0.297 $\pm$ 0.006	0.253 $\pm$ 0.003
concentration 4	0.613 $\pm$ 0.018	0.606 $\pm$ 0.021	0.565 $\pm$ 0.010
concentration 5	0.888 $\pm$ 0.010	0.827 $\pm$ 0.056	0.779 $\pm$ 0.012
<i>Hyalella azteca</i> test	7-d aging	28-d aging	90-d aging
concentration 1	0.022 $\pm$ 0.001	0.021 $\pm$ 0.001	0.022 $\pm$ 0.001
concentration 2	0.040 $\pm$ 0.001	0.043 $\pm$ 0.001	0.047 $\pm$ 0.001
concentration 3	0.076 $\pm$ 0.001	0.067 $\pm$ 0.001	0.066 $\pm$ 0.001
concentration 4	0.143 $\pm$ 0.008	0.141 $\pm$ 0.002	0.151 $\pm$ 0.004
concentration 5	0.291 $\pm$ 0.008	0.279 $\pm$ 0.013	0.284 $\pm$ 0.006

Statistical analysis methods not clearly reported:

“Median lethal sediment concentration (LC50) (nmol/g dw) for permethrin, median lethal tissue residue (LR50) (nmol/g ww lipid), and median lethal fiber concentration (LC50 fiber) (nmol/mL PDMS) estimated by either using standard log-probit analysis (SAS Version 8.02, SAS Institute Cary, NC, USA) or trimmed Spearman-Kärber analyses (TSK), and Abbott’s correction (Abbott 1925) was applied in cases where the data were non-monotonic. Values with overlapping confidence intervals were considered to not be significantly different.”

Reliability points taken off for:

Documentation: Nominal concentrations in interstitial water and/or sediment (2); Overlying water: Source (2), Hardness (1), Alkalinity (1); Significance level (2); % of control at NOEC and/or LOEC (2).

Total: 100-10=90

Acceptability: Control response was within test guidance (10), Measured concentration within 20% nominal (4), \*Spike equilibration time (6), Carrier solvent fully evaporated (4), Organisms randomly assigned to test containers (1), Overlying water source acceptable (2), Hardness within organism tolerance (1), Alkalinity within organism tolerance (1), Random design (2), Adequate replication (2), NOEC response compared to control (1), LOEC response compared to control (1).

\*This is for Sediment 1 (aged 7 d) only.

Total (Sed 2 & 3): 100-29=71

Total (Sed 1)\*: 100-35=65

**Reliability Score: Mean (90, 69) = 80.5 for Sediments 2 & 3 (aged 28 & 90 d, respectively)**

**\* Reliability Score: Mean (90, 63) = 77.5 for Sediment 1 (aged 7 d)**

## ***Appendix 3***

Supplemental data rated N, RN, or LN



## Toxicity Data Summary

### *Chironomus tentans*

Muir DCG, Rawn GP, Townsend BE, Lockhart WL, Greenhalgh R. 1985. Bioconcentration of cypermethrin, deltamethrin, fenvalerate and permethrin by *Chironomus tentans* larvae in sediment and water. Environ Toxicol Chem 4:51-61.

#### Relevance

Score: 60

Rating: N

#### Reliability

Score: n/a

Rating: n/a

\*Reasons for less than 100 pts for relevance: Standard method not reported (10), Chemical purity not stated (15), Toxicity values not calculable (15)

## Toxicity Data Summary

### *Chironomus tentans*

Muir DCG, Rawn GP, Townsend BE, Lockhart WL, Greenhalgh R. 1985. Bioconcentration of cypermethrin, deltamethrin, fenvalerate and permethrin by *Chironomus tentans* larvae in sediment and water. Environ Toxicol Chem 4:51-61.

#### Relevance

Score: 60

Rating: N

#### Reliability

Score: n/a

Rating: n/a

\*Reasons for less than 100 pts for relevance: Standard method not reported (10), Chemical purity not stated (15), Toxicity values not calculable (15)

## Toxicity Data Summary

### *Hexagenia rigida*

Friesen MK, Galloway TD, Flannagan JF (1983) Toxicity of the insecticide permethrin in water and sediment to nymphs of the burrowing mayfly *Hexagenia rigida*. Can Ent 115:1007-1014

#### Relevance

Score: 60

Rating: N

#### Reliability

Score: n/a

Rating: n/a

\*Reasons for less than 100 pts for relevance: Standard method not reported (10), Chemical purity not stated (15), Toxicity values not calculable (15)

## Toxicity Data Summary

### *Chironomus tentans*

Muir DCG, Rawn GP, Townsend BE, Lockhart WL, Greenhalgh R. 1985. Bioconcentration of cypermethrin, deltamethrin, fenvalerate and permethrin by *Chironomus tentans* larvae in sediment and water. Environ Toxicol Chem 4:51-61.

#### Relevance

Score: 60

Rating: N

#### Reliability

Score: n/a

Rating: n/a

\*Reasons for less than 100 pts for relevance: Standard method not reported (10), Chemical purity not stated (15), Toxicity values not calculable (15)